Selective Deprotection of the Diphenylmethylsilyl (DPMS) Hydroxyl Protecting Group Under Environmentally Responsible, Aqueous Conditions

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Supporting Information

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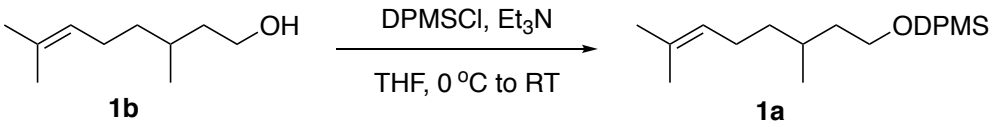
1. General Information

A solution of 2 wt % TPGS-750-M/H_2O was prepared by dissolving TPGS-750-M in degassed HPLC grade water from Fisher Chemical. The resulting solution is stored under argon. The synthesis of TPGS-750-M has been described previously in detail and is available from Sigma-Aldrich (catalog #763896 (wax)). 18-Crown-6 is commercially available from Sigma-Aldrich (catalog #186651). Perfluoro-1-butanesulfonyl fluoride is also commercially available from Sigma-Aldrich (catalog #319732). Certified Grade 1-propanol was purchased from Fisher Chemical. All commercially available starting alcohols were purchased from Sigma-Aldrich or Fisher Chemical. They were used without further purification.

Silica Gel 60 F254 plates (Merck, 0.25nm) were used for thin layer chromatography (TLC). Flash chromatography was done with either an Isolera™ One 3.0 Biotage or a standard glass column using Silica Gel 60 (EMD, 40-63 μm). ^1H and ^{13}C NMR spectra were recorded at 25 °C on either a Varian Unity Inova 500 MHz or a Varian Unity Inova 600 MHz spectrometer in $CDCl_3$ with residual $CHCl_3$ (^1H = 7.26 ppm, ^{13}C = 77.16 ppm) as internal standard. Chemical shifts are reported in parts per million (ppm). The data presented will be reported as follows; chemical shift, multiplicity (s = singlet, bs = broad singlet, d = doublet, dd = doublet of doublet, t = triplet, q = quartet, quin = quintet, m = multiplet), coupling constant (if applicable) and integration. HRMS data were recorded on a Waters Micromass LCT TOF ES+ Premier mass spectrometer using ESI ionization.

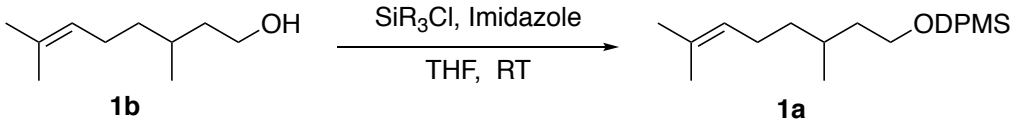
2. General procedure for the protection of DPMS ethers



1b **1a**

All of the alcohols were protected using a modified literature procedure[1]. A round bottom flask under argon was charged with diphenylmethylchlorosilane to which THF was added. The resulting mixture was cooled to 0 °C. Triethylamine was added to the flask followed by a slow addition of a solution of the alcohol in THF over a period of 3 min, resulting in a heterogeneous mixture. The reaction was allowed to warm to rt. The reaction progress was monitored by TLC, and upon completion (3-12 h) the reaction solvent was evaporated under reduced pressure. The resulting crude mixture was diluted with ethyl ether and water. The aqueous layer was extracted with ethyl ether and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure. The crude mixture was purified by flash chromatography.

3. General procedure for the protection of various SiR_3 ethers



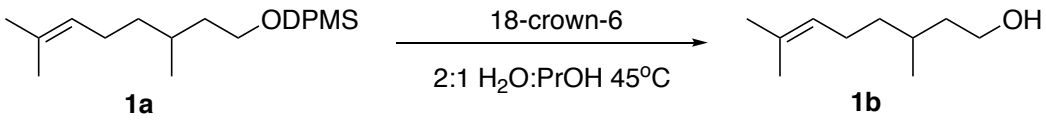
1b **1a**

All other silyl ethers screened for selectivity were synthesized using a modified literature procedure. A round bottom flask under argon was charged with silyl chloride to which was added THF. To the resulting mixture was added imidazole followed by a solution of the alcohol in THF over a period of 3 min, resulting in a heterogeneous mixture. The reaction progress was monitored by TLC, and upon completion (1-6 h) the reaction solvent was evaporated under reduced pressure. The resulting crude mixture was diluted

[1] *J. Org. Chem.* ***2013,*** *9, 2620.*

with ethyl ether and water. The aqueous layer was extracted with ethyl ether and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure. The crude mixture was purified by flash chromatography (10% Et_2O in hexanes).

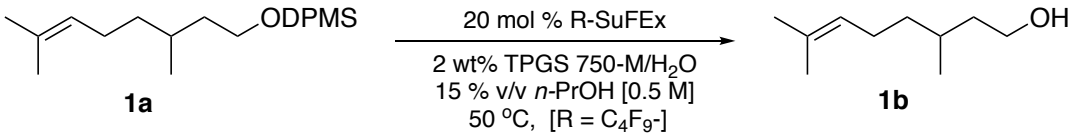
4. Utilization of 18-crown-6 for DPMS ether deprotections



1a **1b**

To a reaction vial equipped with a magnetic stir bar was added 18-crown-6 (53 mg, 0.20 mmol, 1.00 equiv). To this was added ethanol (0.27 mL) and HPLC water (0.53 mL) for a final concentration of 0.25 M. The mixture was stirred at 45 °C giving a clear, homogeneous solution. To this was added **1a** (70.5 mg, 0.20 mmol, 1.00 equiv) dropwise *via* microliter syringe, resulting in a heterogeneous mixture. The reaction progress was monitored by TLC, and upon complete consumption of starting material the vial was brought to rt, then diluted with ethyl ether (2.00 mL). This was shaken and allowed to separate, and the organic layer removed via pipet and filtered over a pad of anhydrous Na_2SO_4 into a round bottom flask. This extraction was repeated an additional two times, and the combined organics were concentrated *in vacuo* to give a crude oil. This was purified by column chromatography (25% diethyl ether in hexanes) to give the pure product.

5. Utilization of perfluoro-1-butanesulfonylfluoride for DPMS ether deprotections



1a **1b**

To a reaction vial equipped with a magnetic stir bar was added the DPMS ether (0.20 mmol, 1 equiv). To this was added a solution of 2 wt % TPGS 750-M/ H_2O (0.85 mL) and propanol (0.15 mL). Perfluoro-1-butanesulfonylfluoride (12.1 mg, 0.04 mmol, 0.20 equiv) was added via microliter syringe resulting in a heterogeneous mixture. The reaction progress was monitored by TLC. Upon complete consumption of starting material (6-24 h), the reaction was allowed to cool to rt, then diluted with ethyl ether (2.0 mL). The vile was shaken and the solution was allowed to separate. This extraction was repeated an additional two times and the combined organics were concentrated *in vacuo* to give a crude oil. This was purified by column chromatography to give the pure product.

6. Recycle study

The initial reaction was set up according to the general procedure (5) stated above. Upon completion of the reaction, the reaction mixture was extracted three times with MTBE in flask (0.6 mL total). The organic extractions were placed in a flask and reduced under pressure. The crude product was purified via a plug of silica with a mixture of EtOAc/hexanes to provide the desired product.

The surfactant solution was then charged with additional DPMS alcohol (0.25 mmol) and 10 mol % perfluoro-1-butanesufonylfluoride. The reaction was sealed and allowed to stir at 50 °C, according to the general procedure.

E Factor Calculation:

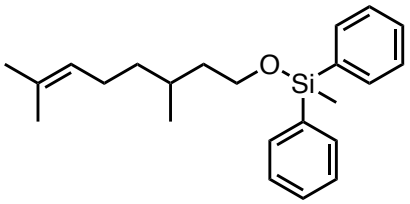
Density of MTBE: 0.74/g/mL

$$E\ Factor = \frac{Waste\ (mg)}{Product\ (mg)}$$

$$E\ Factor = \frac{(0.6mL\ of\ MTBE)\left(\frac{0.74g}{mL}\right)}{0.098g} = 4.5$$

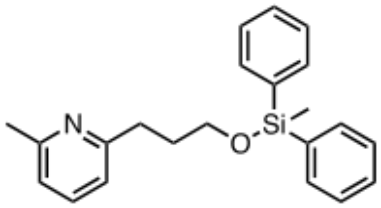
7. Analytical data for silyl ether-protected products

((3,7-Dimethyloct-6-en-1-yl)oxydiphenylmethylsilane (1a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.62-7.59 (dd, 4H), 7.44-7.36 (m, 6H), 5.11-5.06 (m, 1H), 3.79-3.69 (m, 2H), 2.04-1.87 (m, 2H), 1.7 (s, 3H) 1.65-1.55 (m, 2H), 1.60 (s, 3H) 1.44-1.36 (m, 1H) 1.34-1.26 (m, 1H) 1.18-1.10 (m, 1H) 0.85 (d, *J* = 6.6 Hz 3H) 0.65 (s, 3H); **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 136.45, 134.47, 131.20, 129.86, 127.95, 125.00, 61.92, 39.79, 37.30, 29.25, 25.86, 25.59, 19.68, 17.78, -2.88. **Yield**: 89%, 4 h; colorless liquid. **R_f**: 0.80 (25% Et_2O in hexanes). **Chemical Formula**: $C_{23}H_{32}OSi$ EI-MS [M^+] Calcd: 352.2222; found: 337.1988 [$M\text{-}CH_3]^+$

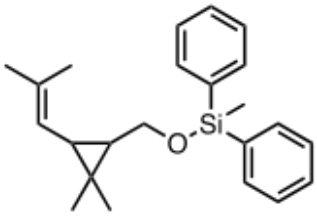
2-Methyl-6-(3-((diphenylmethylsilyl)oxy)propyl)pyridine (2a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.62 – 7.59 (m, 4H), 7.47 – 7.36 (m, 7H), 6.95 (d, *J* = 7.6 Hz, 1H), 6.90 (d, *J* = 7.6 Hz, 1H), 3.77 (t, *J* = 6.4 Hz, 2H), 2.87 – 2.82 (m, 2H), 2.52 (s, 3H), 2.05 – 1.98 (m, 2H), 0.65 (s, 3H).**^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 161.32, 157.86, 136.57, 136.34, 134.49, 129.89, 127.96, 120.54, 119.70, 63.07, 34.90, 32.88, 24.69, -

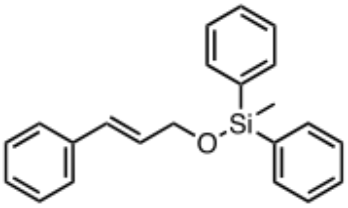
2.91. **Yield**: 91%, 4 h; brown oil. **R_f**: 0.34 (15% EtOAc/hexanes). **Chemical Formula:** $C_{22}H_{25}ONSi$ EI-MS [M^+] Calcd: 347.1705; found: 347.1711

((2,2-Dimethyl-3-(2-methylprop-1-en-1-yl)cyclopropyl)methoxy)diphenylmethylsilyl (3a)



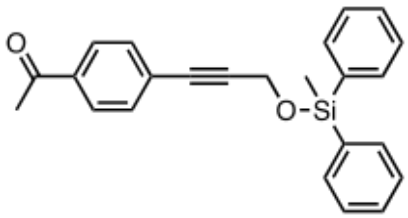
^{1}H NMR (**500 MHz, $CDCl_3$**) major isomer: **δ** 7.61 – 7.59 (m, 4H), 7.40 – 7.36 (m, 6H), 4.85 (ddq, *J* = 9.7, 7.1, 1.4 Hz, 2H), 3.89 (ddd, *J* = 11.2, 6.2, 1.4 Hz, 1H), 3.72 (ddd, *J* = 7.5, 4.0, 1.4 Hz, 1H), 3.63 (ddd, *J* = 11.2, 8.3, 1.3 Hz, 1H), 1.69 (d, *J* = 1.7 Hz, 3H), 1.64 (d, *J* = 1.3 Hz, 3H), 1.07 (d, *J* = 1.4 Hz, 3H), 1.01 (d, *J* = 1.3 Hz, 3H), 0.81 (ddd, *J* = 8.3, 4.2, 1.3 Hz, 1H), 0.64 (d, *J* = 1.4 Hz, 3H).**^{13}C NMR** **(126 MHz, $CDCl_3$**) mixture of isomers: 136.58, 134.66, 134.55, 134.53, 134.14, 132.84, 129.82, 129.80, 127.92, 127.87, 123.89, 119.58, 77.41, 77.16, 76.91, 64.38, 61.30, 34.99, 30.81, 28.95, 28.68, 26.31, 25.86, 25.78, 22.78, 22.45, 21.57, 18.61, 18.39, 15.62, -2.65, -2.71.**Yield**: 85%, 4 h; colorless oil. **R_f:** 0.65 (10% EtOAc/hexanes). **Chemical Formula**: $C_{23}H_{30}OSi$ EI-MS [M^+] Calcd: 350.2065; found: 350.2076.

(Cinnamyloxy)diphenylmethylsilyl (4a)



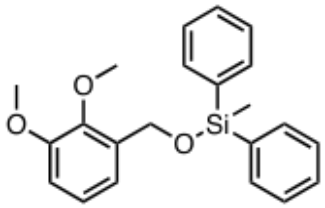
^{1}H NMR (**600 MHz, $CDCl_3$**) **δ** 7.64 – 7.62 (m, 4H), 7.43 – 7.41 (m, 2H), 7.40 – 7.38 (m, 4H), 7.34 (d, *J* = 7.6 Hz, 2H), 7.31 – 7.29 (m, 2H), 7.22 (td, *J* = 7.0, 1.5 Hz, 1H), 6.58 (dd, *J* = 15.9, 1.8 Hz, 1H), 6.31 – 6.27 (m, 1H), 4.42 (dd, *J* = 5.5, 1.8 Hz, 2H), 0.69 (s, *J* = 1.5 Hz, 3H).**^{13}C NMR (151 MHz, $CDCl_3$)** δ 139.76, 138.65, 137.20, 133.09, 132.70, 131.29, 131.19, 130.71, 130.21, 129.23, 67.06, -0.00. **Yield**: 85%, 6 h; colorless oil. **R_f**: 0.33 (15%Et_2O/hexanes). **Chemical Formula**: $C_{22}H_{22}OSi$ EI-MS [M^+] Calcd: 330.1440; found: 330.1425.

1-(4-(3-((Diphenylmethylsilyl)oxy)prop-1-yn-1-yl)phenyl)ethan-1-one (5a)



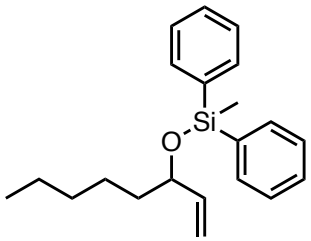
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.90 – 7.86 (m, 2H), 7.69 – 7.66 (m, 4H), 7.41 – 7.39 (m, 8H), 4.63 (s, 2H), 2.59 (s, 3H), 0.77 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 171.11, 137.19, 134.63, 134.13, , 131.84, 130.22, 130.04, 129.70, 128.38, 128.07, 127.86, 51.77, 26.77, -1.08. **Yield**: 69% 3 h; yellow oil. **R_f**: 0.65 (15 % EtOAc/hexanes). **Chemical Formula:** $C_{24}H_{22}O_2Si$ EI-MS [M^+] Calcd: 370.1389; found: 370.1384.

((2,3-Dimethoxybenzyl)oxy)diphenylmethylsilane (6a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.63-7.61 (dd, 4H), 7.45-7.37 (m, 6H), 7.14-7.13 (d, 1H), 7.09-7.05 (t, 1H) 6.86-6.85 (d, 1H) 4.87 (s, 2H), 3.86 (s, 3H), 3.75 (s, 3H), 0.70 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 152.43, 136.11, 134.74, 134.54, 129.97, 129.16, 128.00, 124.07, 120.14, 111.53, 60.75, 60.65, 55.92, -2.86. **Yield**: 96% 4 h; colorless oil. **R_f**: 0.33 (15% Et_2O in hexanes). **Chemical Formula:** $C_{22}H_{24}O_3Si$ EI-MS [M^+] Calcd: 364.1494; found: 364.149

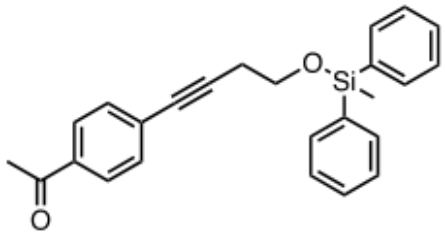
(Oct-1-en-3-yloxy)diphenylmethylsilane (7a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.60 (m, *J* = 6.7, 5.2, 1.5 Hz, 4H), 7.41 – 7.34 (m, 6H), 5.83 (ddd, *J* = 17.0, 10.4, 6.5 Hz, 1H), 5.08 (dt, *J* = 17.2, 1.5 Hz, 1H), 5.01 (ddd, *J* = 10.4, 1.7, 1.1 Hz, 1H), 4.20 – 4.15 (m, 1H), 1.51 – 1.45 (m, 1H), 1.34 – 1.15 (m, 7H), 0.84 (t, *J* = 7.1 Hz, 3H), 0.65 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 141.41, 136.94, 136.86, 134.69, 134.63, 129.86, 129.83, 127.91, 127.89, 114.42, 74.84, 37.98, 31.93, 24.93, 22.79, 14.23,

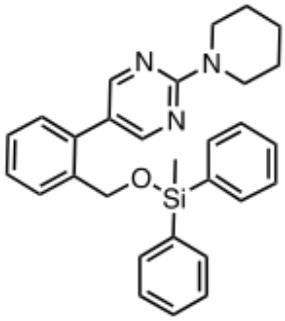
-1.97. **Yield**: 89%, 8 h; colorless liquid. **R_f**: 0.42 (hexanes). **Chemical Formula:** $C_{21}H_{28}OSi$ EI-MS [M^+] Calcd: 324.1909; found: 324.1909.

1-(4-(4-((Diphenylmethylsilyl)oxy)but-1-yn-1-yl)phenyl)ethan-1-one (8a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.87 – 7.84 (m, 2H), 7.62 – 7.59 (m, 4H), 7.43 – 7.34 (m, 8H), 3.91 – 3.88 (t, 2H), 2.70 (t, *J* = 6.9 Hz, 2H), 2.57 (s, 3H), 0.67 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 197.50, 136.01, 135.84, 134.51, 131.86, 130.10, 130.03, 128.85, 128.29, 128.07, 81.35, 62.01, 26.73, 23.87, -2.84. **Yield**: 76%, 4 h; yellow oil. **R_f**: 0.27 (15% EtOAc/hexanes). **Chemical Formula**: $C_{25}H_{24}O_2Si$ EI-MS [M^+] Calcd: 384.1545; found: 384.1546

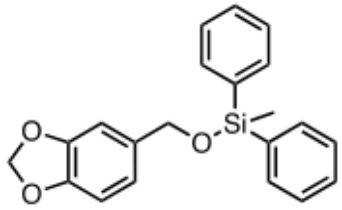
5-(2-(((Diphenylmethylsilyl)oxy)methyl)phenyl)-2-(piperidin-1-yl)pyrimidine (9a)



^{1}H NMR (**600 MHz, $CDCl_3$**) **δ** 8.26 (s, 2H), 7.54 (ddd, *J* = 6.8, 4.0, 1.6 Hz, 5H), 7.40 – 7.36 (m, 2H), 7.33 (qd, *J* = 7.1, 1.3 Hz, 6H), 7.15 (dd, *J* = 7.2, 1.7 Hz, 1H), 4.68 (s, 2H), 3.82 – 3.79 (m, 4H), 1.71 – 1.67 (m, 2H), 1.65 – 1.61 (m, 4H), 0.60 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 160.83, 157.40, 138.38, 135.72, 135.10, 134.39, 129.91, 129.89, 128.72, 127.92, 127.82, 127.70, 121.75, 63.34, 44.94, 25.83, 24.93, -2.94. **Yield**: 85%, 4 h;

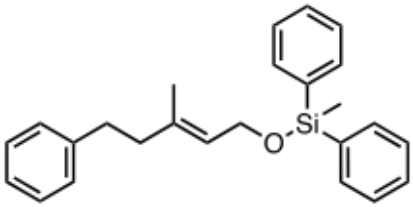
colorless oil. **R_f**: 0.30 (7.5% EtOAc/hexanes). **Chemical Formula:** $C_{29}H_{31}ON_3Si$ EI-MS [M^+] Calcd: 465.2236; found: 466.2315 [M+H]$^+$.

(Benzo[d][1,3]dioxol-5-ylmethoxy)diphenylmethylsilane (10a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.63-7.61 (dd, *J* = 6.4 Hz, 4H), 7.45-7.37 (m, 6H), 6.84 (s, *J* = 0.9 Hz, 1H), 6.75 (d, *J* = 1.0 Hz, 2H), 5.94 (s, 2H), 4.69 (s, *J* = 0.7 Hz, 2H) 0.69 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 147.75, 146.81, 135.93, 134.76, 134.54, 130.05, 128.05, 120.03, 108.12, 107.69, 101.02, 65.39, -2.74. **Yield**: 83%, 4 h; colorless liquid. **R_f**: 0.30 (10% Et_2O/hexanes). **Chemical Formula:** $C_{21}H_{20}O_3Si$ EI-MS [M^+] Calcd: 348.1181; found: 348.1182.

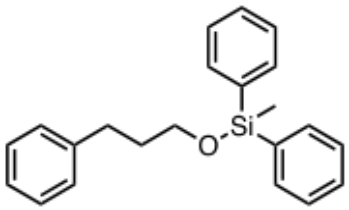
(*E*)-(3-Methyl-5-phenylpent-2-en-1-yl)oxy)diphenylmethylsilane (11a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.62 (dd, *J* = 6.4, 1.6 Hz, 3H), 7.55 – 7.53 (m, 1H), 7.43 – 7.38 (m, 6H), 7.35 – 7.29 (m, 3H), 7.20 (m, 3H), 5.42 (m, 1H), 4.30 – 4.27 (d, 2H), 2.70 (t, *J* = 9.6, 6.9 Hz, 2H), 2.30 – 2.27 (d, 2H), 1.59 (s, 3H), 0.66 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** 142.32, 137.55, 136.33, 134.57, 134.15, 129.90, 128.44, 127.97, 125.90, 124.16, 60.62, 41.51, 34.45, 16.61, -2.57. **Yield**: 78%, 5 h; colorless oil. **R_f**: 0.40 (10%

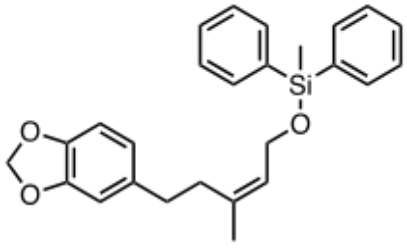
Et_2O/hexanes). **Chemical Formula:** $C_{25}H_{28}O_3Si$ EI-MS [M^+] Calcd: 404.1807; found: 357.1917 [$M\text{-}CH_3]^+$

Diphenylmethyl(3-phenylpropoxy)silane (12a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.65-7.52 (dd, 4H), 7.47-7.39 (m, 6H), 7.30-7.27 (t, 2H), 7.21-7.17 (m, 3H), 3.78-3.75 (t, 2H), 2.75-2.71 (t, *J* = 7.7Hz, 2H), 1.96-1.89 (m, 2H) 0.69 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 142.19, 136.34, 134.48, 129.91, 128.58, 128.00, 127.98, 125.81, 62.86, 34.26, 32.22, -2.90. **Yield**: 87%, 3 h; colorless liquid. **R_f**: 0.80 (25% Et_2O in hexanes). **Chemical Formula:** $C_{22}H_{24}OSi$ EI-MS [M^+] Calcd: 332.1596; found: 317.1361 $[M\text{-}CH_3]^+$

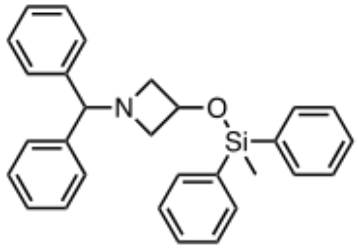
(*Z*)-((5-(Benzo[1,3]dioxol-5-yl)-3-methylpent-2-en-1-yl)oxy)diphenylmethylsilyl (13a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.61 – 7.58 (m, 4H), 7.39 (m, 6H), 6.72 (d, *J* = 7.8 Hz, 1H), 6.66 (d, *J* = 1.7 Hz, 1H), 6.60 (dd, *J* = 8.0, 1.6 Hz, 1H), 5.91 (s, 2H), 5.38 (td, *J* = 6.5, 1.5 Hz, 1H), 4.26 (d, *J* = 6.5 Hz, 2H), 2.62 – 2.58 (m, 2H), 2.22 (dd, *J* = 9.7, 6.5 Hz, 2H), 1.55 (s, 3H), 0.64 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 147.62, 145.68, 137.39, 136.28,

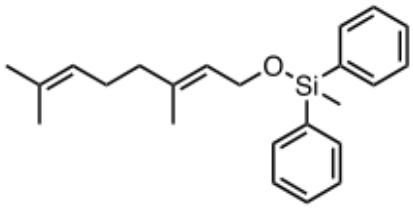
136.15, 134.56, 129.91, 127.96, 124.22, 121.14, 108.95, 108.23, 100.86, 60.59, 41.77, 34.15, 16.58, -2.59. **Yield**: 82%, 4 h; colorless oil. **R_f**: 0.32 (5% EtOAc/hexanes). **Chemical Formula**: $C_{26}H_{28}O_3Si$ EI-MS [M^+] Calcd: 416.1807; found: 439.1700 [M+Na]$^+$

Benzhydryl-3-((diphenylmethylsilyl)oxy)azetidine (14a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.54 (d, *J* = 7.4 Hz, 4H), 7.39 (m, 11H), 7.27 (s, 2H), 7.24 (s, 1H), 7.18 (t, *J* = 7.3 Hz, 2H), 4.53 (p, *J* = 6.2 Hz, 1H), 4.35 (s, 1H), 3.47 (td, *J* = 6.2, 2.3 Hz, 2H), 2.95 (td, *J* = 6.3, 2.3 Hz, 2H), 0.60 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 142.40, 135.74, 134.39, 130.09, 128.52, 128.48, 128.05, 127.58, 127.19, 78.62, 63.55, 62.39, -2.48. **Yield**: 77%, 12 h; colorless oil. **R_f**: 0.32 (15% Et_2O/hexanes). **Chemical Formula**: $C_{29}H_{29}ONSi$ EI-MS [M^+] Calcd: 435.2018; found: 435.2018.

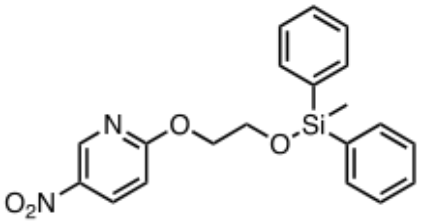
(*E*)-((3,7-Dimethylocta-2,6-dien-1-yl)oxy)diphenylmethylsilyl (15a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.61 (dd, *J* = 6.3, 1.3 Hz, 4H), 7.45 – 7.36 (m, 6H), 5.42 – 5.37 (m, 1H), 5.06 – 5.01 (m, 1H), 4.24 (dt, *J* = 6.7, 1.1 Hz, 2H), 1.97 (tq, *J* = 9.7, 5.0, 3.6 Hz, 4H), 1.71 (s, *J* = 1.2 Hz, 3H), 1.65 (s, *J* = 1.5 Hz, 3H), 1.55 (s, *J* = 3.0 Hz, 3H), 0.66 (s, *J* = 0.9 Hz, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 138.46, 136.29, 134.57, 131.97, 129.88,

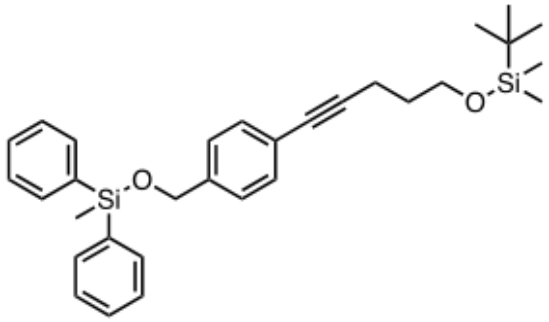
127.96, 124.61, 124.04, 60.27, 32.31, 26.78, 25.80, 23.57, 17.76, -2.59. **Yield**: 81%, 4 h; colorless oil. **R_f**: 0.35 (10% Et_2O/hexanes). **Chemical Formula**: $C_{23}H_{30}OSi$ EI-MS [M^+] Calcd: 350.2065; found: 350.2066

2-(2-((Diphenylmethylsilyl)oxy)ethoxy)-5-nitropyridine (16a)



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 9.01 (d, *J* = 2.8 Hz, 1H), 8.31 (dd, *J* = 9.1, 2.8 Hz, 1H), 7.60 – 7.57 (m, 4H), 7.43 – 7.39 (m, 2H), 7.38 – 7.34 (m, 4H), 6.73 (d, *J* = 9.1 Hz, 1H), 4.57 – 4.54 (m, 2H), 4.08 – 4.06 (m, 2H), 0.67 (s, 3H). **^{13}C NMR** (**126 MHz, $CDCl_3$**) **δ** 167.06, 144.81, 135.74, 134.48, 133.97, 130.10, 128.04, 111.54, 76.91, 68.68, 61.87, -2.82. **Yield**: 79%, 4 h; yellow oil. **R_f**: 0.35 (25% EtOAc/hexanes). **Chemical Formula**: $C_{20}H_{20}O_4N_2Si$ EI-MS [M^+] Calcd: 380.1192; found: 365.0958 [$M\text{-}CH_3$]$^+$

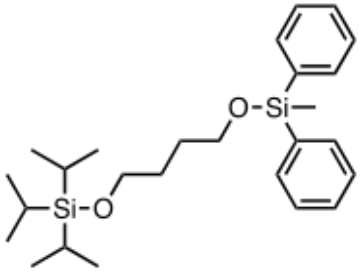
***t*-Butyldimethyl((5-(4-(((diphenylmethylsilyl)oxy)methyl)phenyl)pent-4-yn-1-yl)oxy)silane (17a)**



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.63 – 7.59 (m, 4H), 7.44 – 7.41 (m, 2H), 7.40 – 7.36 (m, 4H), 7.35 – 7.33 (d, 2H), 7.23 (d, *J* = 7.5, 0.8 Hz, 2H), 4.77 (s, 2H), 3.76 (t, *J* = 6.0 Hz, 2H), 2.49 (t, *J* = 7.0 Hz, 2H), 1.83 – 1.78 (m, 2H), 0.91 (s, 9H), 0.65 (s, 3H), 0.08 (s, 6H).

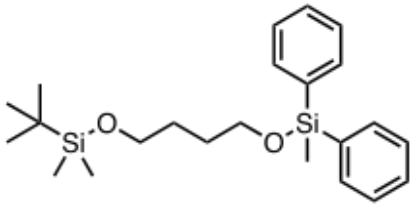
^{13}C NMR (126 MHz, $CDCl_3$) δ 140.21, 135.84, 134.53, 131.58, 130.08, 128.07, 126.39, 105.16, 89.72, 80.81, 77.41, 77.16, 76.90, 65.15, 61.80, 31.93, 26.12, 15.99, -2.78, -5.14. **Yield**: 94%, 4 h; colorless oil. **R_f**: 0.32 (4% EtOAc/hexanes). **Chemical Formula**: $C_{31}H_{40}O_2Si_2$ EI-MS $[M^+]$ Calcd: 500.2566; found: 523.2465 $[M+Na]^+$

9,9-Diisopropyl-10-methyl-2,2-diphenyl-3,8-dioxa-2,9-disilaundecane (18a)



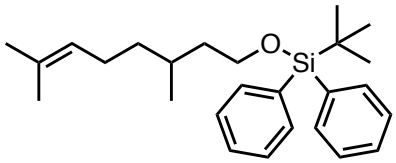
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.59 – 7.53 (m, 4H), 7.39 – 7.32 (m, 6H), 3.69 (dd, *J* = 6.9, 4.4 Hz, 2H), 3.65 (dt, *J* = 6.3, 3.2 Hz, 2H), 1.60 (tt, *J* = 5.2, 2.9 Hz, 4H), 1.04 (s, *J* = 2.4 Hz, 21H), 0.62 (s, *J* = 2.6 Hz, 3H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 134.32, 129.71, 127.80, 63.47, 63.38, 63.18, 29.54, 29.10, 18.03, 12.00, -3.05. **Yield:** 81%, 3 h; light yellow oil. **R_f**: 0.45 (100% hexanes). **Chemical Formula**: $C_{26}H_{42}O_2Si_2$ EI-MS $[M^+]$ Calcd: 442.2723; found: 442.2720.

9,9,10,10-Tetramethyl-2,2-diphenyl-3,8-dioxa-2,9-disilaundecane (19a)



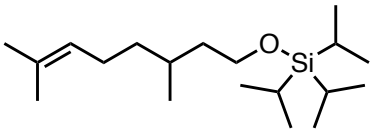
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.57 – 7.55 (m, 4H), 7.38 – 7.33 (m, 6H), 3.69 (t, *J* = 6.3 Hz, 2H), 3.57 (t, *J* = 6.2 Hz, 2H), 1.61 – 1.53 (m, 4H), 0.85 (s, 9H), 0.61 (s, 3H), 0.00 (s, 6H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 136.40, 134.48, 129.88, 127.96, 63.56, 63.14, 29.41, 29.22, 26.12, 18.49, -2.88, -5.13. **Yield:** 77%, 4 h; light yellow oil. **R_f:** 0.42 (100% hexanes). **Chemical Formula**: $C_{23}H_{36}O_2Si_2$ EI-MS $[M^+]$ Calcd: 400.2254; found: 400.2254.

(*t*-Butyl-((3,7-dimethyloct-6-en-1-yl)oxy)diphenylsilane



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.68 (dd, *J* = 7.9, 1.6 Hz, 4H), 7.43 – 7.36 (m, 6H), 5.09 (dddd, *J* = 7.1, 5.7, 2.9, 1.4 Hz, 1H), 3.73 – 3.67 (m, 2H), 1.99 – 1.92 (m, 2H), 1.68 (q, *J* = 1.3 Hz, 3H), 1.64 – 1.59 (m, 5H), 1.38 – 1.29 (m, 2H), 1.16 – 1.11 (m, 1H), 1.05 (s, 9H), 0.84 (d, *J* = 6.5 Hz, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** **δ** 135.57, 134.17, 131.03, 129.47, 127.56, 124.91, 62.20, 39.63, 37.17, 29.05, 26.87, 25.72, 25.49, 19.61, 19.21, 17.65. **Yield**: 68%, 8 h; colorless oil. **R_f**: 0.75 (10% EtOAc/hexanes). Chemical Formula: $C_{26}H_{38}OSi$ EI-MS [M^+] Calcd: 394.2692 found: 394.2692.

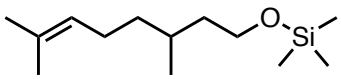
***t*-Butyl-((3,7-dimethyloct-6-en-1-yl)oxy)triisopropylsilane**



^{1}H NMR (**600 MHz, $CDCl_3$**) **δ** 5.10 (m, 1H), 3.74 – 3.68 (m, 2H), 2.02 – 1.93 (m, 2H), 1.68 (d, *J* = 1.4 Hz, 3H), 1.61 – 1.57 (m, 6H), 1.37 – 1.31 (m, 2H), 1.16 (dtd, *J* = 9.5, 5.9, 5.1, 2.1 Hz, 1H), 1.07 – 1.05 (m, 20H), 0.89 (d, *J* = 6.5 Hz, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** **δ** 131.19, 125.08, 105.18, 61.84, 40.24, 37.41, 29.30, 25.67, 19.85, 18.20, 17.78, 12.19.

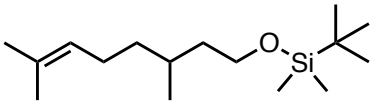
Yield: 94%, 4 h; colorless oil. **R_f**: 0.75 (10% EtOAc/hexanes). **Chemical Formula**: $C_{19}H_{40}OSi$ EI-MS [M^+] Calcd: 312.6130; found: 269.2297 [$M-C_3H_7$]$^+$.

((3,7-Dimethyloct-6-en-1-yl)oxy)trimethylsilane



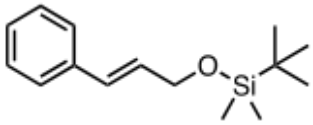
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 5.09 (ddq, *J* = 8.4, 5.5, 1.4 Hz, 1H), 3.64 – 3.57 (m, 2H), 1.97 (tq, *J* = 14.8, 7.6 Hz, 2H), 1.68 (s, *J* = 1.5 Hz, 3H), 1.60 (s, *J* = 1.3 Hz, 3H), 1.58 – 1.50 (m, 2H), 1.37 – 1.30 (m, 2H), 1.19 – 1.12 (m, 1H), 0.88 (d, *J* = 6.6 Hz, 3H), 0.11 (s, 9H). **^{13}C NMR** **(126 MHz, $CDCl_3$)** 131.25, 125.01, 61.07, 39.96, 37.38, 29.35, 25.87, 25.62, 19.74, 17.78, -0.30. **Yield:** 79%, 4 h **R_f:** 0.75 (10% EtOAc/hexanes).

t-Butyl-((3,7-dimethyloct-6-en-1-yl)oxy)dimethylsilane



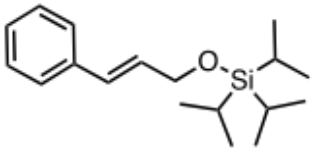
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 5.10 (tp, *J* = 7.0, 1.4 Hz, 1H), 3.67 – 3.60 (m, 2H), 2.02 – 1.93 (m, 4H), 1.68 (s, 3H), 1.60 (s, 3H), 1.57 – 1.54 (m, 3H), 1.33 (m, 2H), 1.19 – 1.12 (m, 1H), 0.89 (s, 9H), 0.05 (s, 6H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 131.22, 125.05, 61.62, 40.10, 37.37, 29.28, 26.13, 25.87, 25.65, 19.79, 18.50, 17.79, -5.11. **Yield:** 84%, 4 h; colorless oil. **R_f**: 0.75 (10% EtOAc/hexanes). **Chemical Formula**: $C_{16}H_{34}OSi$ EI-MS [M^+] Calcd: 270.2379; found: 270.2376.

_t_-Butyl(cinnamyloxy)dimethylsilane



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.38 – 7.34 (m, 2H), 7.31 – 7.26 (m, 2H), 7.22 – 7.18 (m, 1H), 6.57 (dd, *J* = 15.7, 2.0 Hz, 1H), 6.27 (dtd, *J* = 15.9, 5.1, 0.9 Hz, 1H), 4.34 (dt, *J* = 5.1, 1.2 Hz, 2H), 0.93 (d, *J* = 0.9 Hz, 9H), 0.10 (d, *J* = 0.9 Hz, 6H). **δ ^{13}C NMR (126 MHz, $CDCl_3$) δ** 133.36129.46, 129.18, 128.49, 127.29, 126.37, 63.89, 25.98, 18.47, -5.13. **Yield**: 75%, 3 h; colorless liquid. **R_f**: 0.30 (15% Et_2O/hexanes).

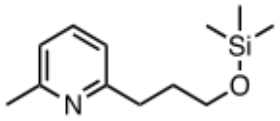
(Cinnamyloxy)triisopropylsilane



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.40 – 7.37 (m, 2H), 7.33 – 7.29 (m, 2H), 7.24 – 7.20 (m, 1H), 6.65 (dd, *J* = 15.7, 2.1 Hz, 1H), 6.33 – 6.27 (m, 1H), 4.44 (dd, *J* = 4.8, 1.9, 0.8 Hz, 2H), 1.20 – 1.14 (m, 3H), 1.12 – 1.09 (d, 18H). **δ ^{13}C NMR (126 MHz, $CDCl_3$) δ** 137.41, 129.52, 129.19, 128.63, 127.35, 126.52, 64.07, 18.20, 12.24. **Yield**: 89%, 3 h; colorless

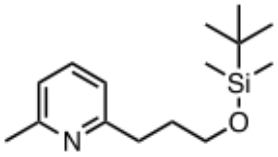
liquid **R_f**: 0.75 (5% EtOAc/hexanes). **Chemical Formula:** $C_{13}H_{30}OSi$ EI-MS [M^+] Calcd. 290.2066 found: 290.2065.

2-Methyl-6-(3-((trimethylsilyl)oxy)propyl)pyridine



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.36 (t, *J* = 7.7 Hz, 1H), 6.85 (dd, *J* = 7.7, 2.7 Hz, 2H), 3.53 (t, *J* = 6.5 Hz, 2H), 2.71 – 2.67 (m, 2H), 2.41 (s, 3H), 1.86 – 1.82 (m, 2H), -0.01 (s, 9H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 161.70, 158.17, 136.92, 120.88, 62.53, 35.24, 33.31, 26.44, 24.98, -0.00. **Yield**: 86% 4 h; light yellow oil. **R_f**: 0.34 (10% EtOAc/hexanes). **Chemical Formiula**: $C_{12}H_{21}NOSi$ EI-MS [M^+] Calcd: 223.1392 found: 223.1391.

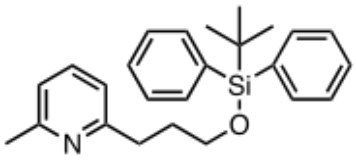
2-Methyl-6-(3-((*t*-butyldimethylsilyl)oxy)propyl)pyridine



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.46 (t, *J* = 7.6 Hz, 1H), 6.95 (dd, *J* = 7.6, 2.2 Hz, 2H), 3.66 (t, *J* = 6.4 Hz, 2H), 2.83 – 2.79 (m, 2H), 2.52 (s, 3H), 1.96 – 1.90 (m, 2H), 0.90 (s, 9H), 0.04 (s, 6H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 161.72, 158.19, 136.90, 120.86, 119.98,

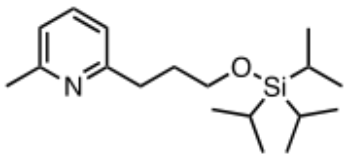
62.55, 35.25, 33.31, 25.00, 16.98, -0.00, -2.31. **Yield**: 89% 4 h; light yellow oil. **Rf**: 0.36 (10% EtOAc/hexanes). **Chemical Formula:** $C_{15}H_{27}NOSi$ EI-MS [M^+] Calcd: 265.1862; found: 265.1867.

2-Methyl-6-(3-((*t*-butyldiphenylsilyl)oxy)propyl)pyridine



^{1}H NMR (**600 MHz, $CDCl_3$**) **δ** 7.71 – 7.68 (m, 1H), 7.65 – 7.63 (m, 4H), 7.38 – 7.33 (m, 5H), 7.24 (s, 1H), 6.91 (dd, *J* = 14.2, 7.6 Hz, 2H), 3.70 (t, *J* = 6.3 Hz, 2H), 2.85 – 2.82 (m, 2H), 2.49 (s, 3H), 1.99 – 1.94 (m, 2H), 1.04 (s, 9H). **^{13}C NMR (151 MHz, $CDCl_3$) δ** 161.27, 157.68, 136.42, 134.77, 133.95, 129.59, 127.67, 120.38, 119.57, 63.30, 34.70, 32.75, 26.85, 24.49, 19.21. **Yield**: 83%, 12 h; light yellow oil. **R_f**: 0.32 (10% EtOAc/hexanes). **Chemical Formula**: $C_{25}H_{31}NOSi$ EI-MS [M^+] Calcd: 389.2175; found: 389.2171.

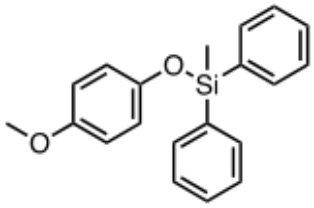
2-Methyl-6-(3-((triisopropylsilyl)oxy)propyl)pyridine



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.44 (t, *J* = 7.6 Hz, 1H), 6.93 (dd, *J* = 9.7, 7.7 Hz, 2H), 3.71 (t, *J* = 6.4 Hz, 2H), 2.84 – 2.79 (m, 2H), 2.50 (s, 3H), 1.96 – 1.91 (m, 2H), 1.03 (d, *J* = 3.9 Hz, 18H).**^{13}C NMR (126 MHz, $CDCl_3$) δ** 161.45, 157.68, 136.42, 120.36, 119.62, 62.78, 34.75, 33.23, 24.49, 18.03, 12.01. **Yield**: 90%, 4 h; light yellow oil. **R_f**: 0.28

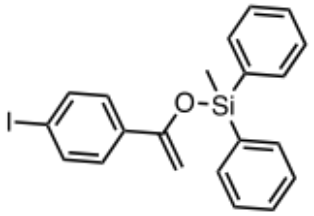
(10% EtOAc/hexanes). **Chemical Formula**: $C_{18}H_{33}ONSi$ EI-MS [M^+] Calcd: 307.2332; found: 307.2344.

4-((Diphenylmethylsilyl)oxy)anisole



^{1}H NMR (**400 MHz, $CDCl_3$**) **δ** 7.69 – 7.65 (m, 4H), 7.46 – 7.39 (m, 6H), 6.79 – 6.71 (m, 4H), 3.73 (s, 3H), 0.75 (s, 3H). **^{13}C NMR (151 MHz, $CDCl_3$) δ** 155.89, 150.41, 137.22, 136.04, 131.73, 129.62, 122.19, 116.12, 57.21, -1.01. **Yield**: 83% 6 h; colorless oil. **R_f**: 0.31 (5% EtOAc/hexanes). **Chemical Formula:** $C_{20}H_{20}O_2Si$ EI-MS [M^+] Calcd: 320.1232; found: 320.1235.

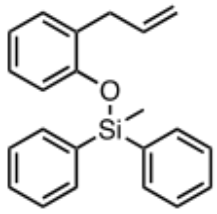
((1-(4-Iodophenyl)vinyl)oxy)diphenylmethylsilane



^{1}H NMR (**600 MHz, C_6D_6**) **δ** 7.65 – 7.63 (m, 4H), 7.40 – 7.37 (m, 2H), 7.21 – 7.19 (m, 2H), 7.19 – 7.17 (m, 6H), 4.71 (d, *J* = 2.2 Hz, 1H), 4.41 (d, *J* = 2.2 Hz, 1H), 0.65 (s, 3H). **^{13}C**

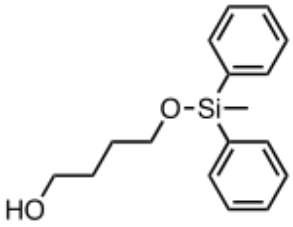
NMR (151 MHz, C_6D_6) δ 154.70, 137.22, 136.92, 135.25, 134.26, 130.03, 127.95, 127.00, 94.03, 92.35, -3.24. **Yield**: 78%, 6 h; colorless oil. **R_f**: 0.30 (5% Et_2O/hexanes). **Chemical Formula:** $C_{21}H_{19}OISi$ EI-MS [M^+] Calcd: 442.0249; found: 442.0233.

(2-Allylphenoxy)diphenylmethylsilane



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.66 (dt, *J* = 6.6, 1.5 Hz, 4H), 7.47 – 7.37 (m, 6H), 7.14 (dd, *J* = 7.5, 1.7 Hz, 1H), 6.97 (td, *J* = 7.7, 1.8 Hz, 1H), 6.89 (td, *J* = 7.4, 1.3 Hz, 1H), 6.69 (dd, *J* = 8.0, 1.3 Hz, 1H), 5.97 (ddtd, *J* = 16.8, 10.2, 6.6, 1.4 Hz, 1H), 5.05 – 4.99 (m, 2H), 3.42 (dt, *J* = 6.7, 1.5 Hz, 2H), 0.76 (s, 3H). **^{13}C NMR (151 MHz, $CDCl_3$) δ** 155.89, 150.41, 137.22, 136.04, 131.73, 130.33, 129.62, 122.19, 116.12, 57.21, -1.01. **Yield**: 76%, 6 h; colorless oil. **R_f**: 0.30 (5% Et_2O/hexanes). **Chemical Formula**: $C_{22}H_{22}OSi$ EI-MS [M^+] Calcd: 330.1439; found: 330.1431.

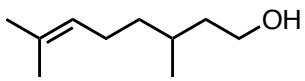
4-((Methyldiphenylsilyl)oxy)butan-1-ol



^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.61 – 7.57 (m, 4H), 7.44 – 7.36 (m, 6H), 3.76 – 3.73 (m, 2H), 3.65 – 3.61 (m, 2H), 1.68 – 1.64 (m, 4H), 0.65 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 135.81, 134.33, 129.89, 127.90, 63.45, 62.78, 29.75, 29.26, -3.12. **Yield:** 85%, 2 h; light yellow oil. **R_f**: 0.26 (25% EtOAc/hexanes). **Chemical Formula:** $C_{17}H_{22}O_2Si$ EI-MS [M^+] Calcd: 286.1389; found: 286.1390.

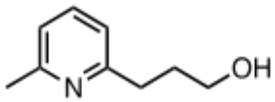
8. Analytical data for free alcohols

3,7-Dimethyloct-6-en-1-ol (1b)



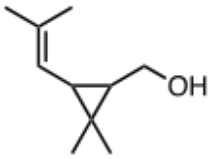
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 5.14 – 5.08 (m, 1H), 3.70 (m, 2H), 2.00 (tq, *J* = 14.8, 7.5 Hz, 2H), 1.70 (d, *J* = 3.0 Hz, 3H), 1.62 (d, *J* = 2.8 Hz, 3H), 1.37 (dtdd, *J* = 21.2, 8.4, 5.1, 2.6 Hz, 3H), 1.21 (tdd, *J* = 13.6, 6.7, 3.4 Hz, 2H), 0.92 (dd, *J* = 6.5, 2.7 Hz, 3H). **^{13}C NMR** **(126 MHz, $CDCl_3$)** **δ** 131.25, 124.69, 61.20, 37.21, 29.17, 25.70, 25.45, 19.52, 17.63. **Yield**: 88% 12 h (SFx), 91% 2.5 h (18-crown-6), colorless oil. **R_f:** 0.35 (50% Et_2O/hexanes).

3-(6-Methyl-2-pyridinyl)-1-propanol (2b)



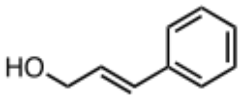
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.52 – 7.48 (m, 1H), 6.98 (d, *J* = 7.7 Hz, 2H), 3.72 (td, *J* = 5.6, 2.8 Hz, 2H), 2.94 (td, *J* = 6.9, 2.9 Hz, 2H), 2.51 (d, *J* = 2.8 Hz, 3H), 1.97 (dtq, *J* = 8.7, 5.9, 3.3, 2.5 Hz, 2H). **^{13}C NMR** **(126 MHz, $CDCl_3$)** **δ** 160.69, 157.30, 137.06, 120.02, 62.35, 61.91, 35.63, 31.55, 24.07. **Yield:** 94% 6 h (SFx), 90% 3 h (18-crown-6), reddish brown oil. **R_f:** 0.25 (65% EtOAc/hexanes).

[2,2-Dimethyl-3-(2-methyl-1-propen-1-yl)cyclopropyl]metanol (3b)



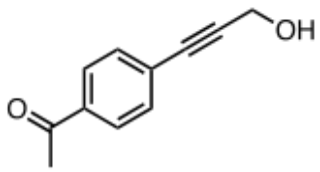
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** major isomer 4.87 (1H, d, *J* = 8.1), 3.77 (1H, dd, *J* = 6.6, 11.4), 3.55 (1H, dd, *J* = 8.5, 11.4), 1.70 (3H, s,), 1.67 (3H, s,), 1.15 (3H, s), 1.11 (1H, dd, *J* = 5.3, 8.1), 1.06 (3H, s), 0.83 (1H, ddd, *J* = 8.5, 6.6, 5.3); minor isomer: 4.96 (1H, d, *J* = 8.2), 3.67 (1H, dd, *J* = 7.6, 11.6), 3.61 (1H, dd, *J* = 8.0, 11.6), 1.73 (3H, s), 1.70 (3H, s), 1.38 (1H, dd, *J* = 8.2), 1.12 (3H, s), 1.07–1.04 (1H, m) 1.04 (3H, s) **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** major isomer: 133.0, 123.5, 63.5, 35.1, 28.6, 25.6, 22.7, 21.3, 18.3, 15.5; minor isomer 135.0, 119.1, 60.4, 31.0, 28.8, 26.2, 25.8, 22.3, 20.8, 18.4. **Yield**: 94% 16 h (SFx), 94% 3 h (18-crown-6).

(2*E*)-3-Phenyl-2-propen-1-ol (4b)



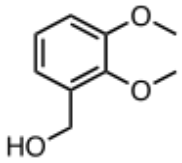
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.39 – 7.35 (m, 2H), 7.31 (dd, *J* = 8.5, 6.8 Hz, 2H), 7.25 – 7.21 (m, 1H), 6.60 (dt, *J* = 15.9, 1.6 Hz, 1H), 6.35 (dt, *J* = 15.9, 5.8 Hz, 1H), 4.31 (dd, *J* = 5.7, 1.6 Hz, 2H), 1.77 – 1.66 (m, 1H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 136.71, 131.07, 128.55, 127.69, 126.49, 126.48, 63.63. **Yield**: 93%, 8 h (SFx), 85%, 2 h (18-crown-6), white solid. **R_f**: 0.40 (25% EtOAc/hexanes).

1-(4-(3-Hydroxyprop-1-yn-1-yl)phenyl)ethan-1-one (5b)



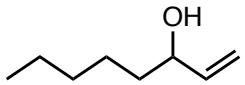
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.92 – 7.89 (m, 2H), 7.53 – 7.49 (m, 2H), 4.53 (d, *J* = 6.2 Hz, 2H), 2.60 (s, 3H), 1.75 (t, *J* = 6.2 Hz, 1H). **^{13}C NMR** (**126 MHz, $CDCl_3$**) **δ** 197.46, 136.63, 131.94, 128.38, 127.55, 90.62, 85.06, 51.78, 26.78. **Yield**: 79%, 12 h (SFx), 88%, 6 h (18-crown-6), ; red oil. **R_f**: 0.37 (50% EtOAc/hexanes).

(2,3-Dimethoxyphenyl)metanol (6b)



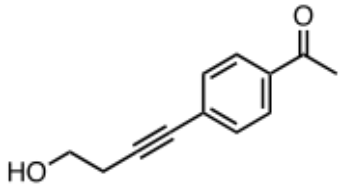
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.03 (td, *J* = 7.9, 1.5 Hz, 1H), 6.92 – 6.86 (m, 2H), 4.68 (dd, *J* = 6.4, 1.5 Hz, 2H), 3.87 (d, *J* = 1.4 Hz, 3H), 3.86 (d, *J* = 1.5 Hz, 3H), 2.18 (tdd, *J* = 6.3, 4.1, 2.3 Hz, 1H). **^{13}C NMR** (**126 MHz, $CDCl_3$**) **δ** 152.50, 134.58, 124.18, 120.64, 112.23, 61.56, 60.87, 55.81. **Yield**: 83% 12 h (SFx), 93% 2.5 h (18-crown-6), white solid. **R_f**: 0.32 (25% EtOAc/hexanes).

1-Octen-3-ol (7b)



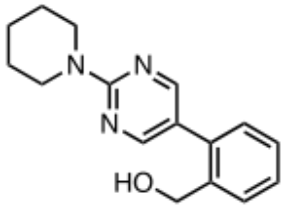
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 5.85 (ddd, *J* = 16.9, 10.4, 6.2 Hz, 1H), 5.20 (dt, *J* = 17.2, 1.4 Hz, 1H), 5.08 (dt, *J* = 10.4, 1.4 Hz, 1H), 4.07 (tdd, *J* = 7.4, 5.5, 1.3 Hz, 1H), 1.70 – 1.54 (m, 1H), 1.54 – 1.44 (m, 2H), 1.38 (dddd, *J* = 13.6, 8.5, 6.6, 3.0 Hz, 1H), 1.34 – 1.23 (m, 5H), 0.89 – 0.84 (m, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 141.32, 114.46, 73.24, 36.97, 31.74, 24.99, 22.58, 14.00. **Yield:** 68% 36 h (SFx), 75% 22 h (18-crown-6); colorless liquid. **R_f**: 0.35 (25% EtOAc/hexanes).

1-(4-(4-Hydroxybut-1-yn-1-yl)phenyl)ethan-1-one (8b)



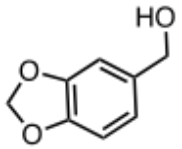
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.88 – 7.86 (d, 2H), 7.49 – 7.45 (d, 2H), 3.82 (q, *J* = 6.2 Hz, 2H), 2.71 (t, *J* = 6.2 Hz, 2H), 2.57 (s, 3H), 1.75 (t, *J* = 6.3 Hz, 1H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 196.55, 131.80, 128.31, 128.19, 104.99, 90.16, 81.82, 61.03, 26.61, 23.93. **Yield:** 79% 12 h (SFx), 93% 4 h (18-Crown-6) tan solid. **R_f**: 0.35 (80% Et_2O/hexanes). **Chemical Formula**: $C_{12}H_{12}O_2$ EI-MS $[M^+]$ Calcd: 188.0837; found: 189.0913 $[M+H]^+$.

(2-(2-(Piperidin-1-yl)pyrimidin-5-yl)phenyl)metanol (9b)



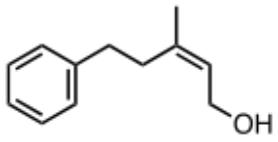
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 8.33 (s, 2H), 7.55 – 7.52 (m, 1H), 7.39 – 7.32 (m, 2H), 7.21 – 7.19 (m, 1H), 4.61 (d, *J* = 4.3 Hz, 2H), 3.82 – 3.79 (m, 4H), 1.99 (d, *J* = 5.0 Hz, 1H), 1.68 (dddd, *J* = 8.3, 6.9, 3.9, 2.2 Hz, 2H), 1.64 – 1.59 (m, 4H). **^{13}C NMR** (**126 MHz, $CDCl_3$**) **δ** 160.83, 148.91, 137.72, 137.10, 129.91, 129.89, 128.72, 127.92, 127.82, 63.34, 44.94, 25.83, 24.93. **Yield**: 89% 14 h (SFx), 86% 8 h (18-crown-6). colorless oil. **R_f:** 0.27 (60 EtOAc in hexanes). **Chemical Formula**: $C_{16}H_{19}ON_3$ EI-MS [M^+] Calcd: 269.1528; found: 270.1608 $[M+H]^+$.

Benzo[*d*][1,3]dioxol-5-ylmethanol (10b)



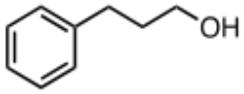
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 6.86 (s, *J* = 1.5 Hz, 1H), 6.79 (m, *J* = 7.9, 6.1 Hz, 2H), 5.95 (s, *J* = 1.9 Hz, 2H), 4.57 (s, *J* = 3.4 Hz, 2H). **^{13}C NMR** (**126 MHz, $CDCl_3$**) **δ** 147.90, 135.00, 120.59, 108.30, 107.98, 101.10, 100.97, 65.29, **Yield:** 92% 12 h(SFx), 83% 1 h (18-crown-6), 85%; white solid. **R_f**: 0.33 (40% Et_2O/hexanes).

(2*E*)-3-Methyl-5-phenyl-2-penten-1-ol (11b)



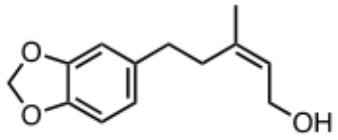
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.32 – 7.27 (m, 2H), 7.22 – 7.18 (m, 3H), 5.43 (tp, *J* = 6.9, 1.3 Hz, 1H), 4.16 (d, *J* = 6.9 Hz, 2H), 2.79 – 2.74 (m, 2H), 2.35 (dd, *J* = 9.5, 6.7 Hz, 2H), 1.75 (s, 3H), 1.28 – 1.16 (m, 1H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 141.98, 132.21, 128.37, 128.32, 125.85, 123.90, 59.28, 41.38, 34.34, 16.40. **Yield:** 87% 16 h (SFx), 92% 3 h (18-crown-6), yellow oil. **R_f:** 0.26 (25% EtOAc/hexanes).

3-Phenyl-1-propanol (12b)



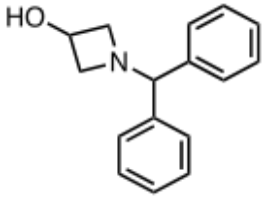
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.31 – 7.27 (m, 2H), 7.21 (d, *J* = 7.7 Hz, 3H), 3.68 (td, *J* = 6.5, 1.2 Hz, 2H), 2.74 – 2.69 (m, 2H), 1.90 (dtd, *J* = 9.0, 7.6, 7.0, 5.8 Hz, 2H), 1.55 (d, *J* = 7.1 Hz, 1H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 141.85, 128.44, 128.41, 125.87, 62.04, 34.16, 32.09. **Yield:** 89% 14 h (SFx), 94% 2.5 h (18-crown-6), colorless oil. **R_f:** 0.31 (25% EtOAc/hexanes).

(*Z*)-5-(Benzo[1,3]dioxol-5-yl)-3-methylpent-2-en-1-ol (13b)



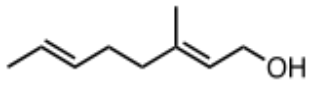
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 6.72 (d, *J* = 7.9 Hz, 1H), 6.67 (d, *J* = 1.7 Hz, 1H), 6.61 (dd, *J* = 7.8, 1.7 Hz, 1H), 5.91 (s, 2H), 5.41 (m, 1H), 4.14 (d, *J* = 6.9 Hz, 2H), 2.68 – 2.63 (m, 2H), 2.28 (dd, *J* = 8.9, 7.2 Hz, 2H), 1.71 (s, 3H), 1.25 (s, 1H). **^{13}C NMR (126 MHz, $CDCl_3$)** **δ** 147.65, 145.73, 139.17, 135.95, 124.08, 121.31, 108.94, 108.23, 100.89, 59.48, 41.78, 34.21, 16.53. Yield: 90% 16 h (SFx), 92% 4 h (18-crown-6); yellow oil. **R_f**: 0.40 (25% EtOAc/hexanes).

1-(Diphenylmethyl)-3-azetidinol (14b)



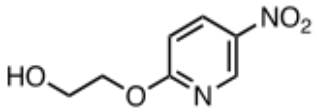
^{1}H NMR (**600 MHz, $CDCl_3$**) **δ** 7.39 – 7.36 (m, 4H), 7.26 (t, *J* = 7.7 Hz, 4H), 7.19 – 7.16 (m, 2H), 4.44 (p, *J* = 5.8 Hz, 1H), 4.33 (s, 1H), 3.54 – 3.51 (m, 2H), 2.90 – 2.87 (m, 2H). **^{13}C NMR (151 MHz, $CDCl_3$)** **δ** 141.87, 128.44, 127.41, 127.16, 78.45, 63.36, 62.08, **Yield:** 80% 24 h (SFx), 78% 20 h (18-crown-6), white solid. **R_f:** 0.29 (25% EtOAc/hexanes).

(Z)-3,7-Dimethylocta-2,6-dien-1-ol (15b)



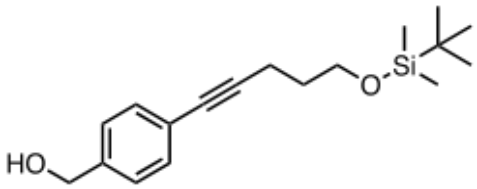
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 5.42 (td, *J* = 7.2, 1.5 Hz, 1H), 5.08 (m, 1H), 4.07 (d, *J* = 7.2 Hz, 2H), 2.11 – 2.03 (m, 4H), 1.73 (d, *J* = 1.3 Hz, 3H, 1.58 (s, 3H), 1.17 (d, *J* = 6.1 Hz, 1H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 132.31, 124.47, 123.81, 58.86, 31.95, 26.53, 25.61, 23.37, 17.60. **Yield:** 85% 6 h (SFx), 91% 3 h (18-crown-6), tan oil. **R_f:** 0.36 (25% EtOAc/hexanes).

2-((5-Nitropyridin-2-yl)oxy)ethan-1-ol (16b)



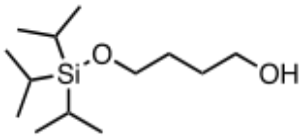
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 9.06 (d, *J* = 2.8 Hz, 1H), 8.38 (dd, *J* = 9.1, 2.9 Hz, 1H), 6.89 (d, *J* = 8.6 Hz, 1H), 4.59 – 4.57 (m, 2H), 4.02 – 3.99 (m, 2H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 167.06, 144.73, 139.81, 134.30, 111.64, 69.46, , 61.52. **Yield:** 86% 5 h (SFx), 79% 6 h (18-crown-6), white solid. **R_f**: 0.35 (80% EtOAc/hexanes).

(4-(5-(*t*-Butyldimethylsilyl)oxy)pent-1-yn-1-yl)phenyl)methanol (17b)



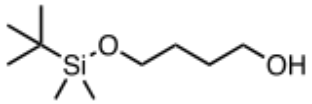
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 7.40 – 7.37 (m, 2H), 7.29 – 7.26 (m, 2H), 4.68 (d, *J* = 5.9 Hz, 2H), 3.76 (t, *J* = 6.0 Hz, 2H), 2.49 (t, *J* = 7.0 Hz, 2H), 1.84 – 1.78 (m, 2H), 1.67 (t, *J* = 6.0 Hz, 1H), 0.91 (s, 9H), 0.08 (s, 6H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 140.30, 131.85, 126.90, 123.49, 90.16, 80.63, 65.20, 61.79, 31.89, 26.12, 18.53, 15.99, -5.15. **Yield:** 71% 16 h (SFx), 80% 8 h (18-crown-6), 81%; brown oil. **R_f:** 0.35 (15% EtOAc/hexanes). **Chemical Formula**: $C_{18}H_{30}O_2$ EI-MS [M^+] Calcd: 278.2245; found: 247.1154 [$M-C_4H_9$]$^+$.

4-((Triisopropylsilyl)oxy)butan-1-ol (18b)



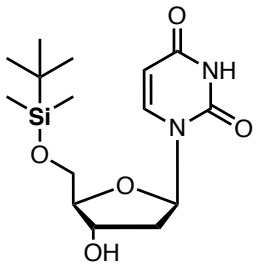
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 3.73 (q, 2H), 3.64 (q, 2H), 2.54 (s, 1H), 1.69 – 1.59 (m, 5H), 1.05 (d, 18H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 63.59, 62.85, 30.35, 30.06, 17.96, 11.94. **Yield:** 82% 12 h (SFx) 89% 3 h (18-crown-6); colorless oil. **R_f**: 0.28 (25% EtOAc/hexanes).

4-((*t*-Butyldimethylsilyl)oxy)butan-1-ol (19b)



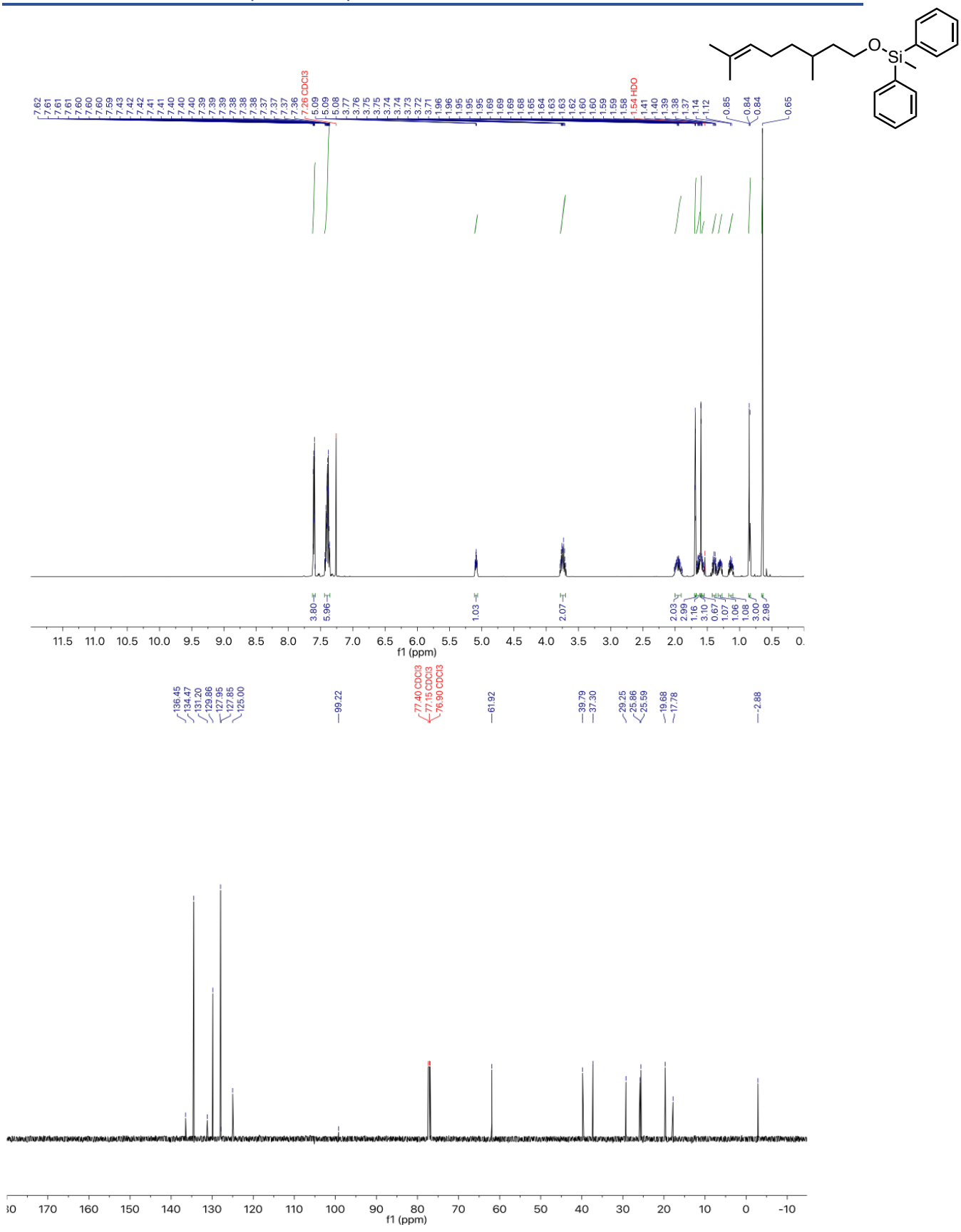
^{1}H NMR (**500 MHz, $CDCl_3$**) **δ** 3.63 (dt, *J* = 12.4, 5.7 Hz, 4H), 1.65 – 1.60 (m, 4H), 0.88 (s, 9H), 0.05 (s, 6H). **^{13}C NMR (126 MHz, $CDCl_3$) δ** 63.33, 62.77, 30.23, 29.86, 25.89, 18.29, -5.40. **Yield:** 90% 12 h (SFx), 86% 3 h (18-crown-6); colorless oil. **R_f:** 0.25 (25% EtOAc/hexanes).

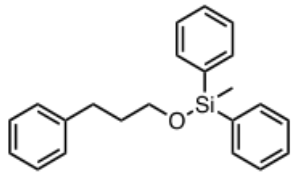
5-(((*tert*-butyldimethylsilyl)oxy)methyl)-4-hydroxytetrahydrofuran-2-yl)pyrimidine-2,4(1*H*,3*H*)-dione (20b)

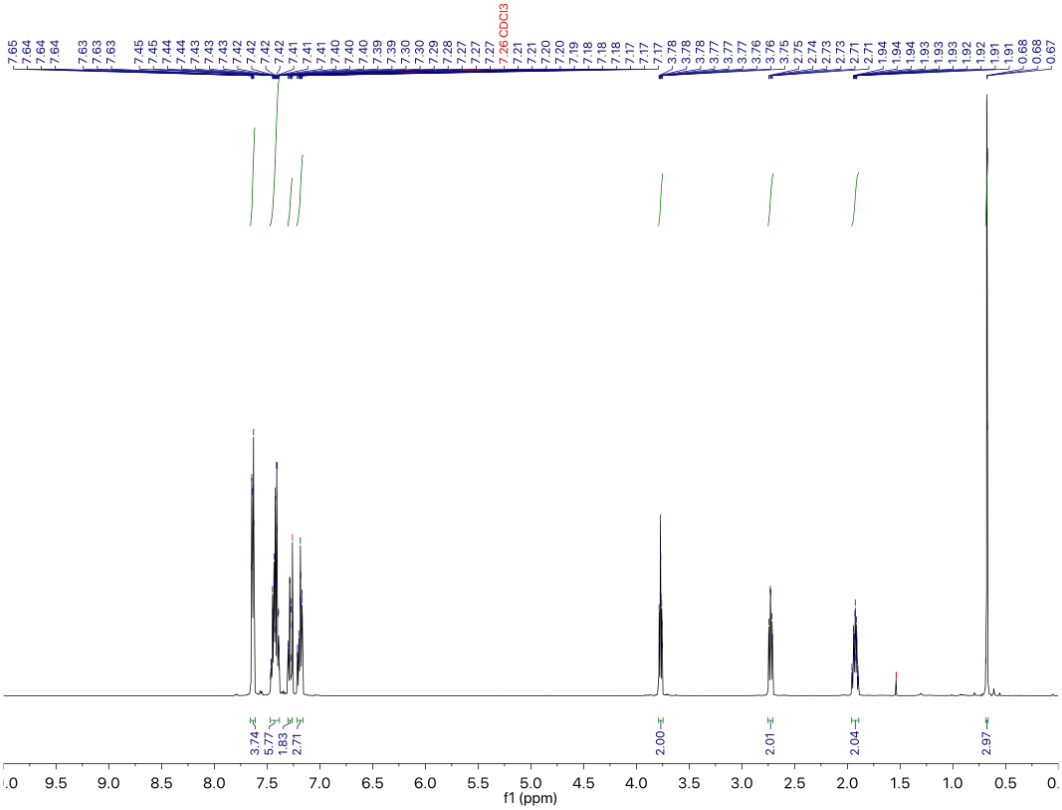


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.91 (d, *J* = 8.1 Hz, 1H), 6.38 – 6.33 (m, 1H), 5.69 (d, *J* = 8.1 Hz, 1H), 4.46 (dt, *J* = 5.9, 3.0 Hz, 1H), 4.05 (q, *J* = 2.5 Hz, 1H), 3.90 (dd, *J* = 11.4, 2.6 Hz, 1H), 3.82 (dd, *J* = 11.4, 2.2 Hz, 1H), 2.42 (m, 1H), 2.17 (s, 1H), 2.16 – 2.09 (m, 1H), 0.90 (s, 9H), 0.09 (s, 6H). **^{13}C NMR (101 MHz, $cdcl_3$) δ** 150.63, 140.37, 102.46, 87.51, 85.49, 77.48, 72.21, 63.46, 41.67, 26.00, 18.46, -5.36. **Yield**: 86% 10 h (SFx), 81% 8 h (18-crown-6); colorless oil. **R_f**: 0.24 (70% EtOAc/hexanes).

9. ^{1}H NMR and ^{13}C NMR spectra for products



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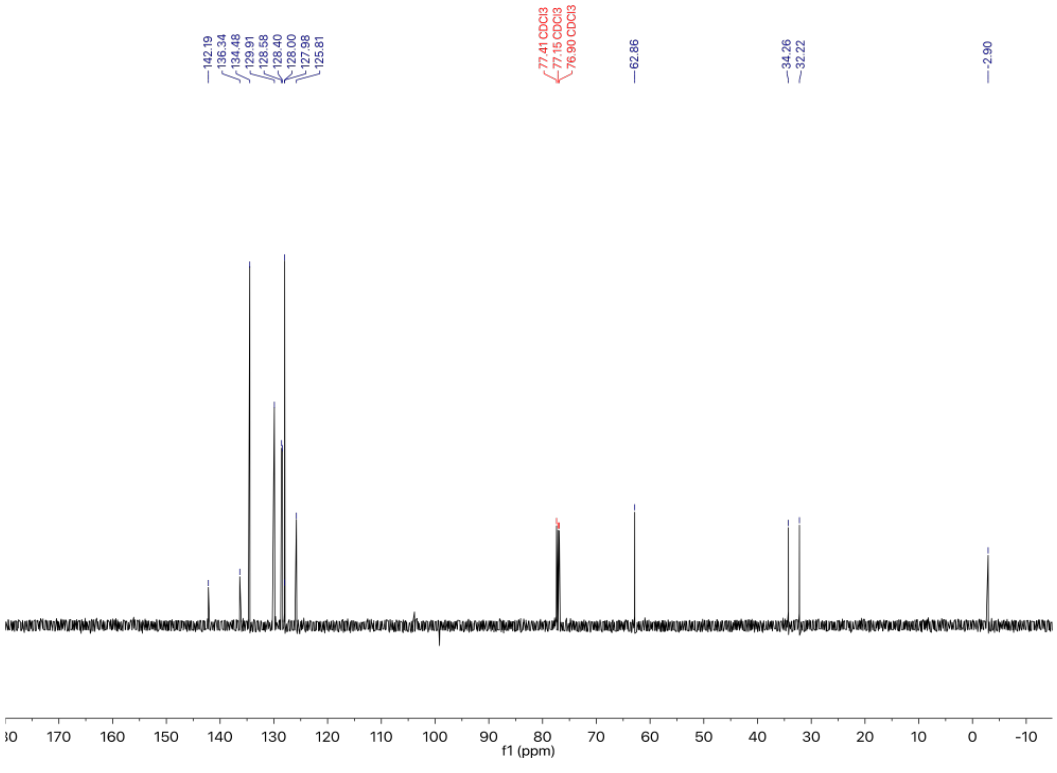
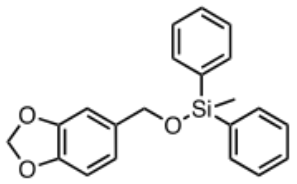
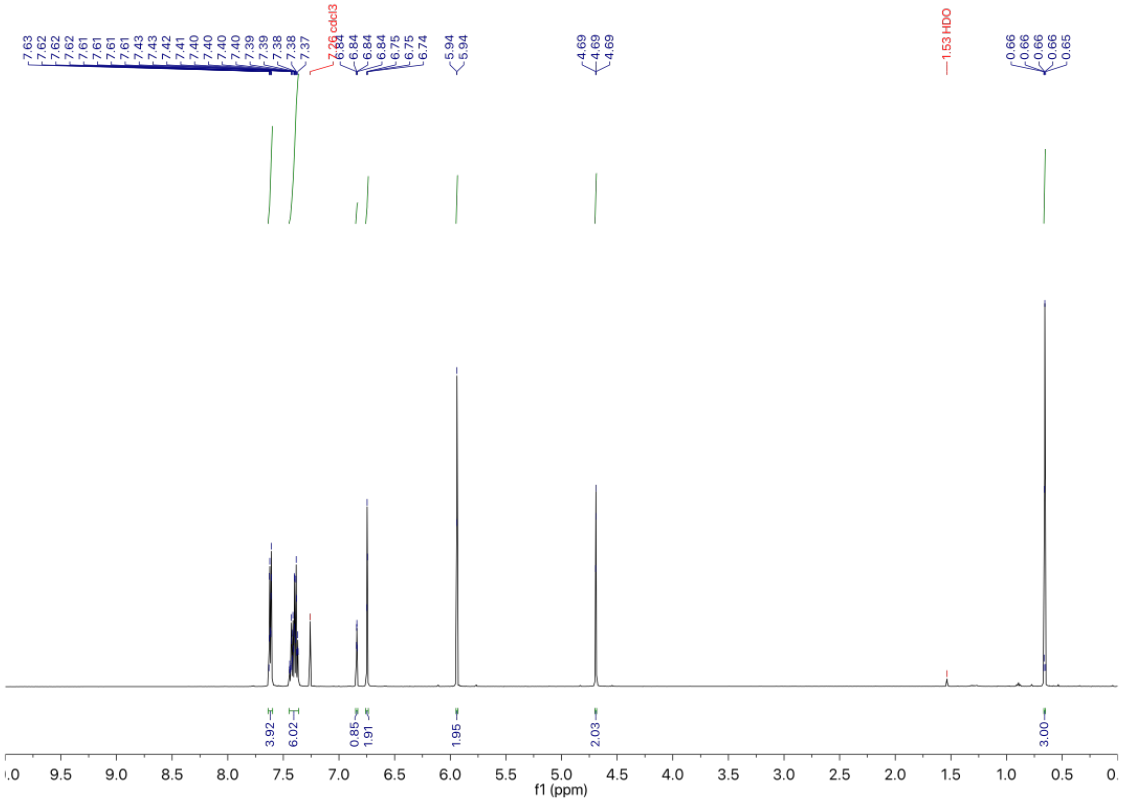
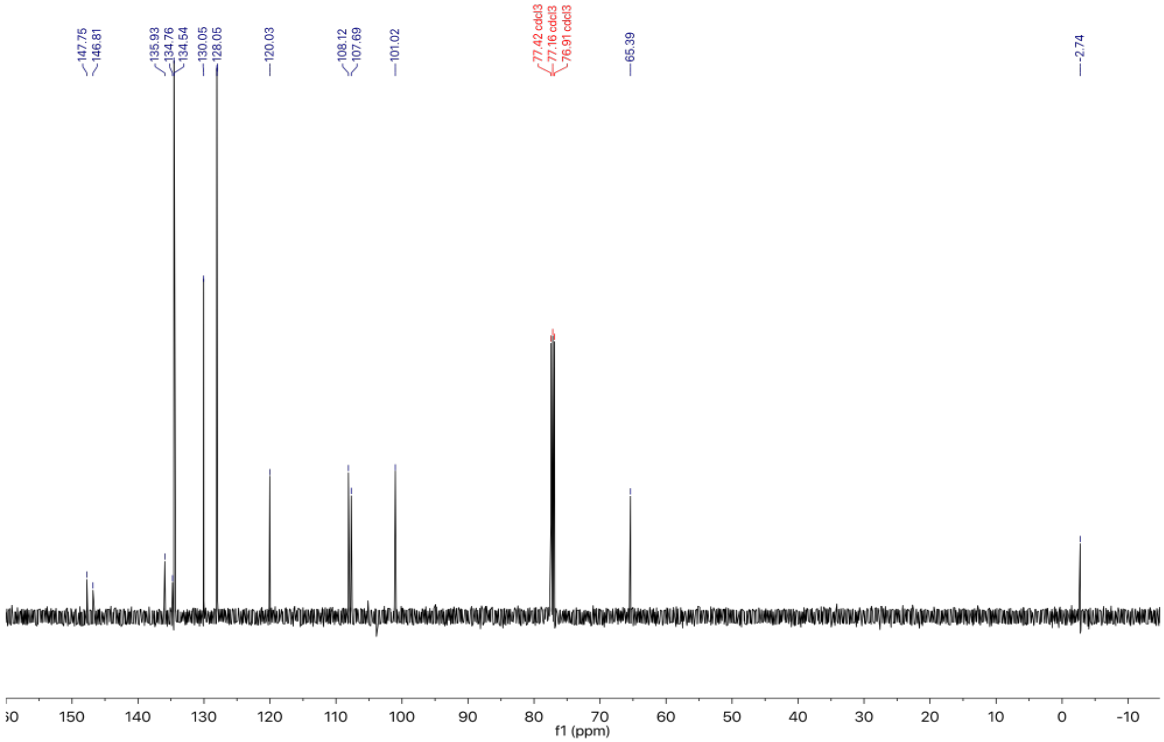
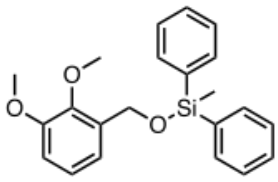
3.74
5.77
1.83
2.71
2.00
2.01
2.04
2.97
f1 (ppm)

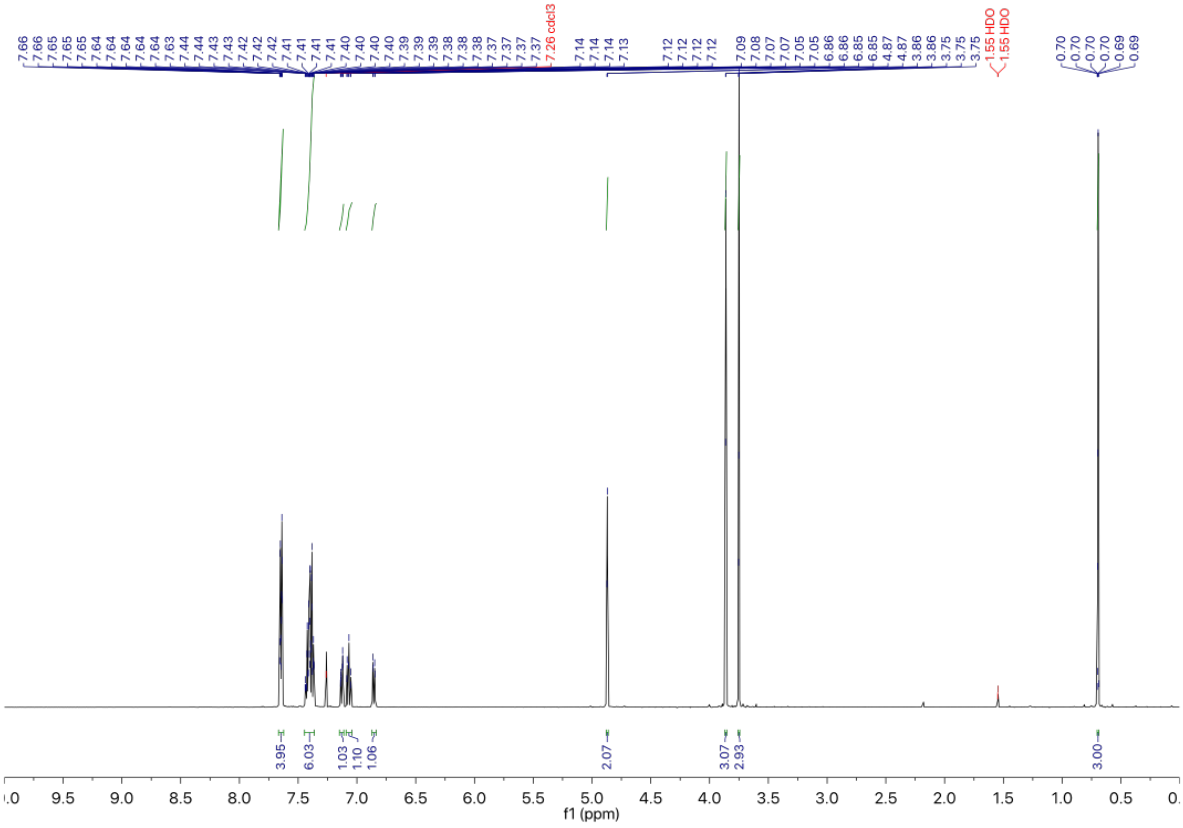

142.19
136.34
134.48
129.91
128.58
128.40
128.00
127.98
125.81
77.41 CDCl3
77.15 CDCl3
76.90 CDCl3
62.86
34.26
32.22
-2.90
f1 (ppm)


O
O
O
Si


7.63
7.62
7.62
7.62
7.61
7.61
7.61
7.61
7.43
7.43
7.42
7.41
7.40
7.40
7.40
7.40
7.39
7.39
7.38
7.38
7.37
7.26 cdcl3
6.84
6.84
6.84
6.84
6.75
6.75
6.74
5.94
5.94
4.69
4.69
4.69
1.53 HDO
0.66
0.66
0.66
0.66
0.66
0.65
3.92
6.02
0.85
1.91
1.95
2.03
3.00
f1 (ppm)


147.75
146.81
135.93
134.76
134.54
130.05
128.05
120.03
108.12
107.69
101.02
77.42 cdcl3
77.16 cdcl3
76.91 cdcl3
65.39
-2.74
f1 (ppm)




7.26 cdcl3
1.55 HDO
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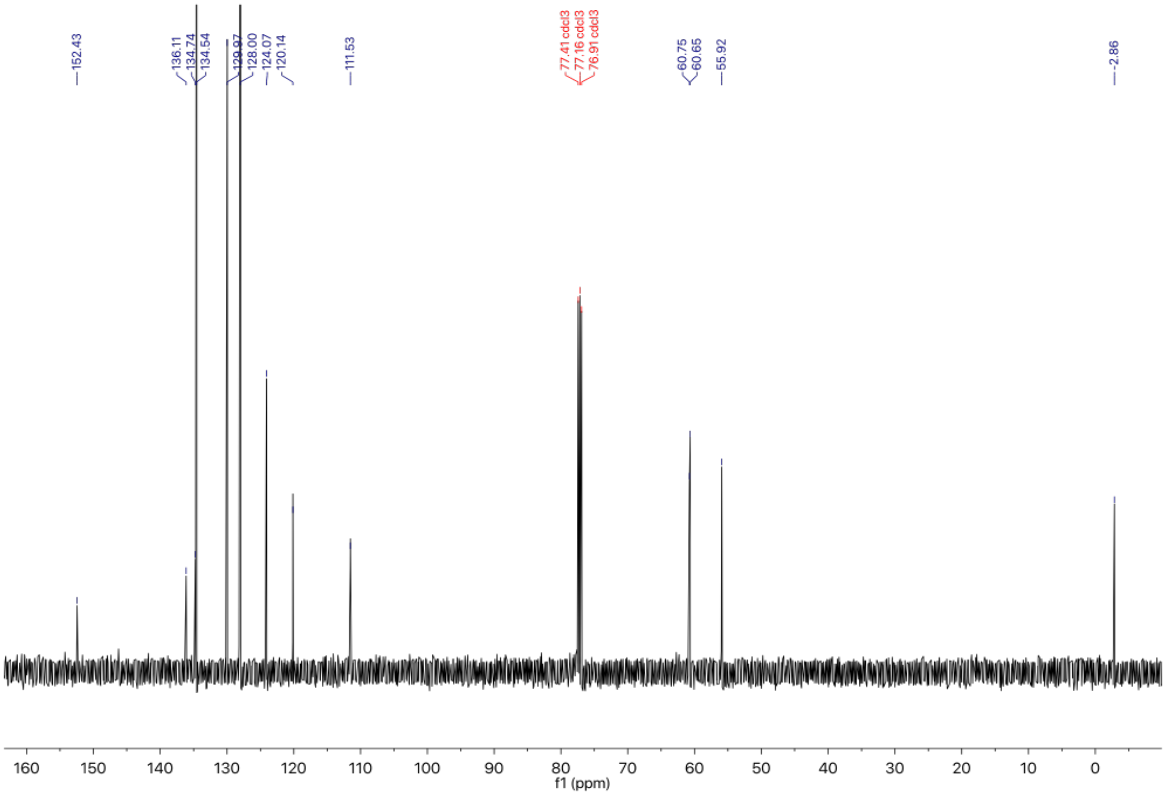
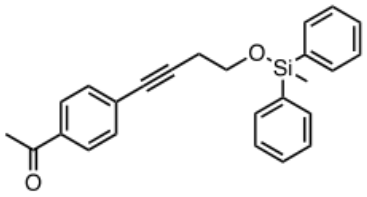
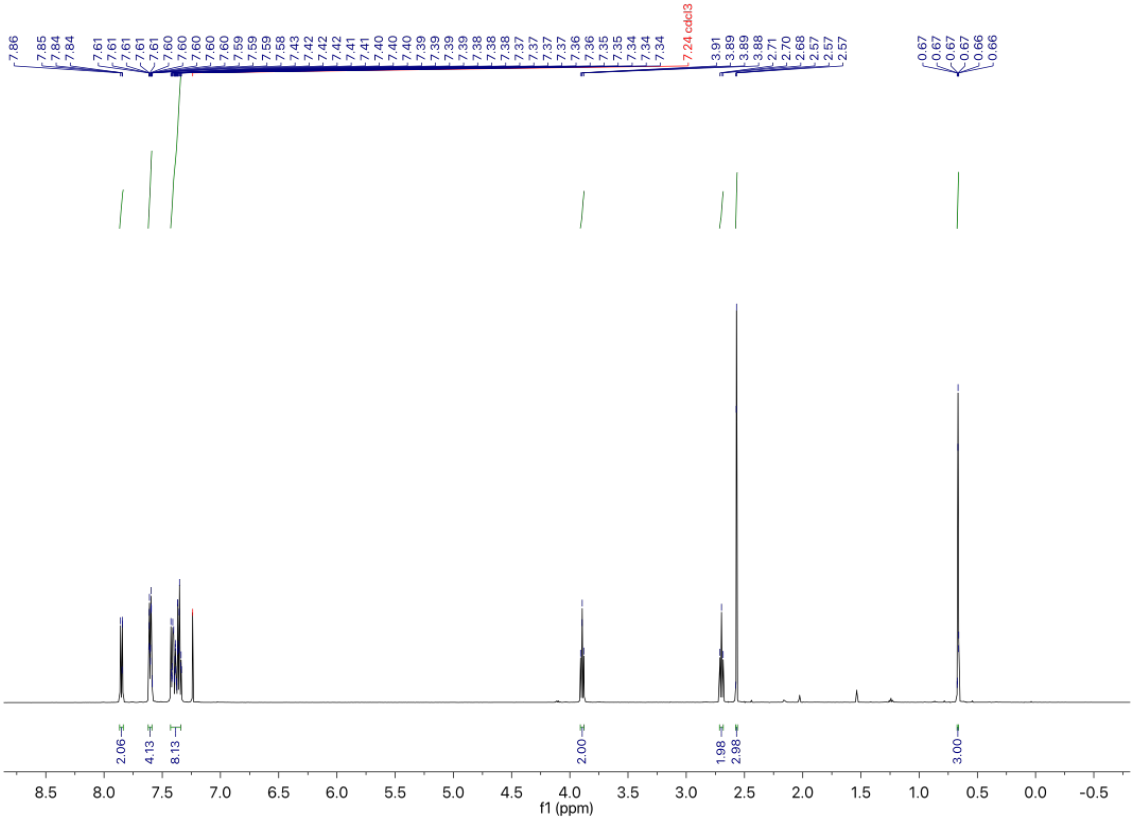
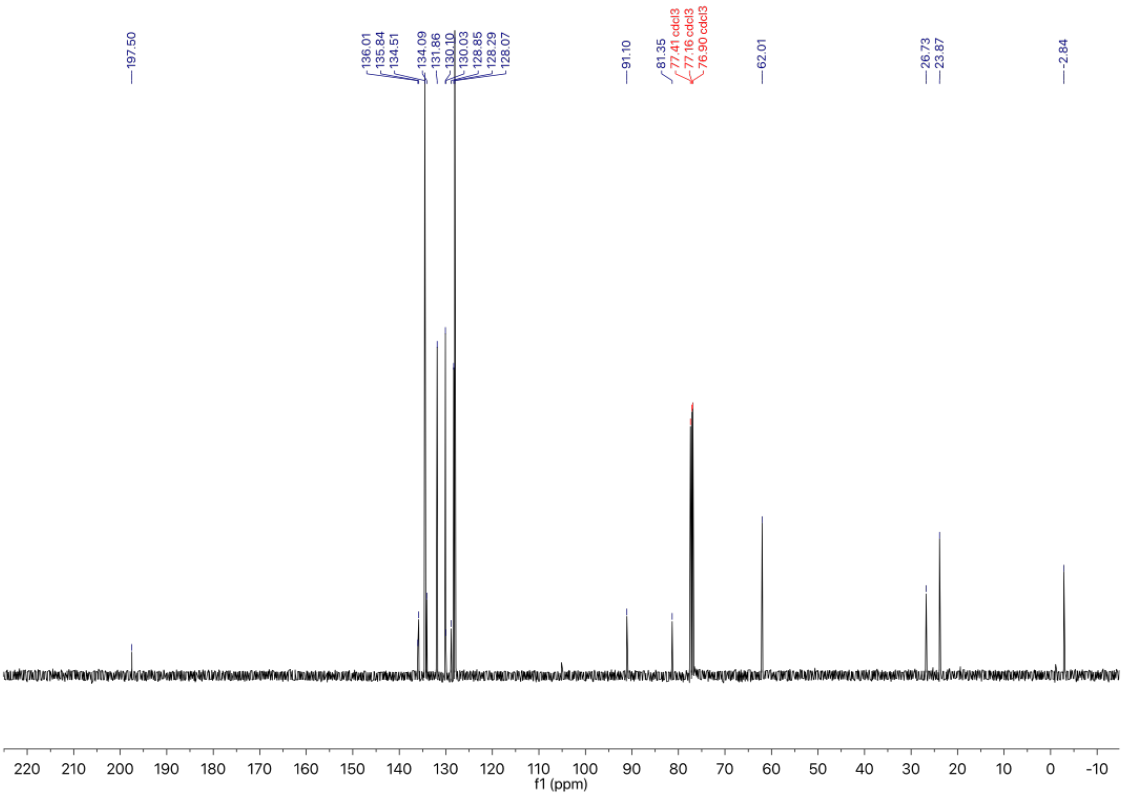
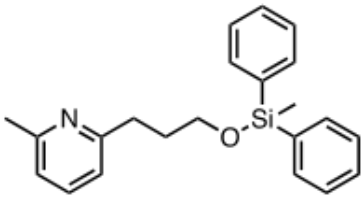
6.03
1.03
1.10
1.06
2.07
3.07
2.93
3.00
f1 (ppm)

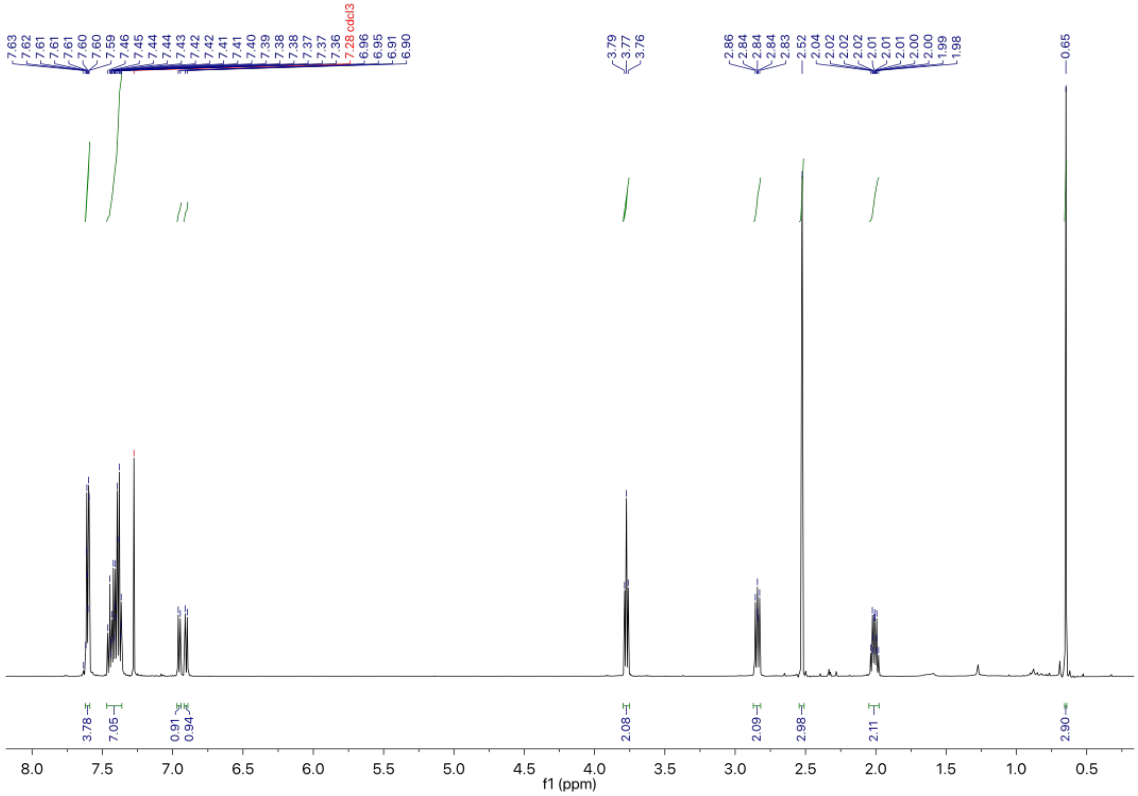

152.43
136.11
134.74
134.54
129.97
128.00
124.07
120.14
111.53
77.41 cdcl3
77.16 cdcl3
76.91 cdcl3
60.75
60.65
55.92
-2.86
f1 (ppm)


O
Si
O


7.86 7.85 7.84 7.84 7.61 7.61 7.61 7.61 7.60 7.60 7.60 7.60 7.60 7.60 7.59 7.59 7.59 7.58 7.43 7.42 7.42 7.42 7.41 7.41 7.40 7.40 7.40 7.39 7.39 7.39 7.39 7.38 7.38 7.38 7.37 7.37 7.37 7.37 7.36 7.36 7.35 7.35 7.34 7.34 7.34 7.24 cdcl3 3.91 3.89 3.89 3.89 3.88 2.71 2.70 2.68 2.57 2.57 2.57 0.67 0.67 0.67 0.67 0.67 0.66 0.66
2.06 4.13 8.13 2.00 1.98 2.98 3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


197.50 136.01 135.84 134.51 134.09 131.86 130.10 130.03 128.85 128.29 128.07 91.10 81.35 77.41 cdcl3 77.16 cdcl3 76.90 cdcl3 62.01 26.73 23.87 -2.84
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


N
O
Si


7.63
7.62
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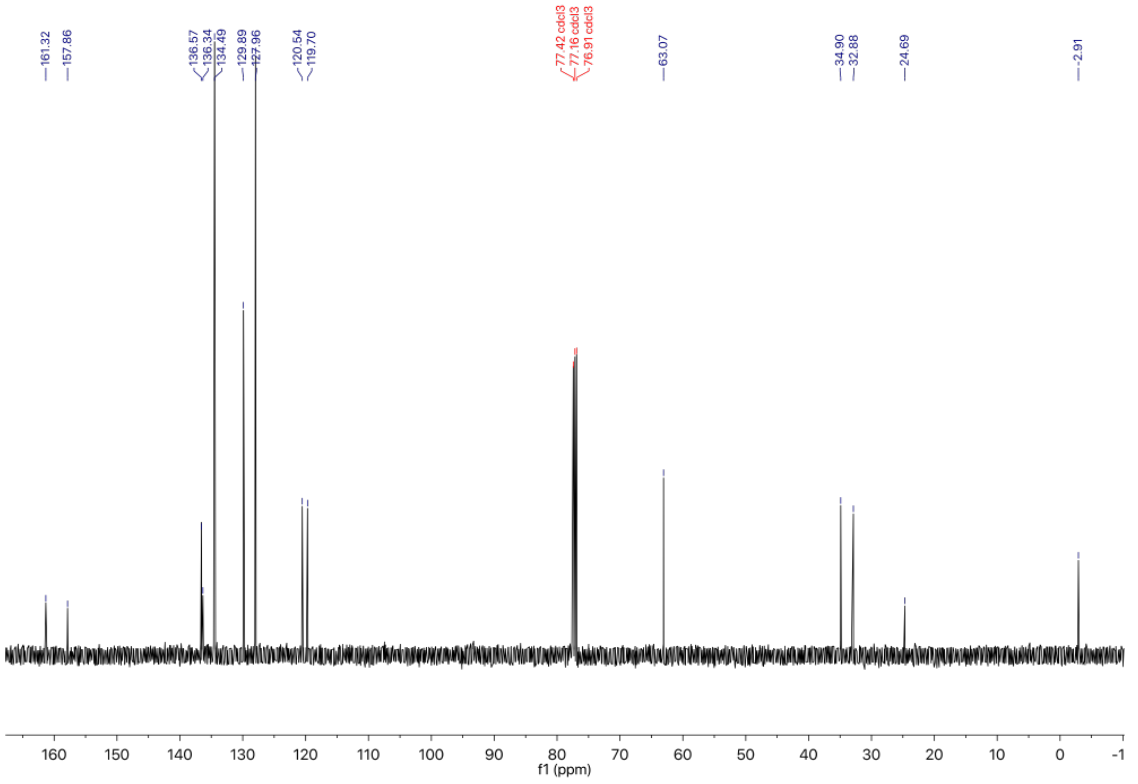
2.52
2.04
2.02
2.02
2.02
2.01
2.01
2.01
2.00
2.00
1.99
1.98
0.65
3.78
7.05
0.91
0.94
2.08
2.09
2.98
2.11
2.90
f1 (ppm)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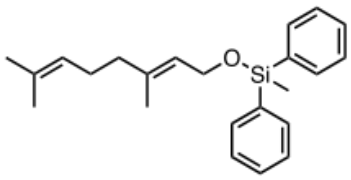
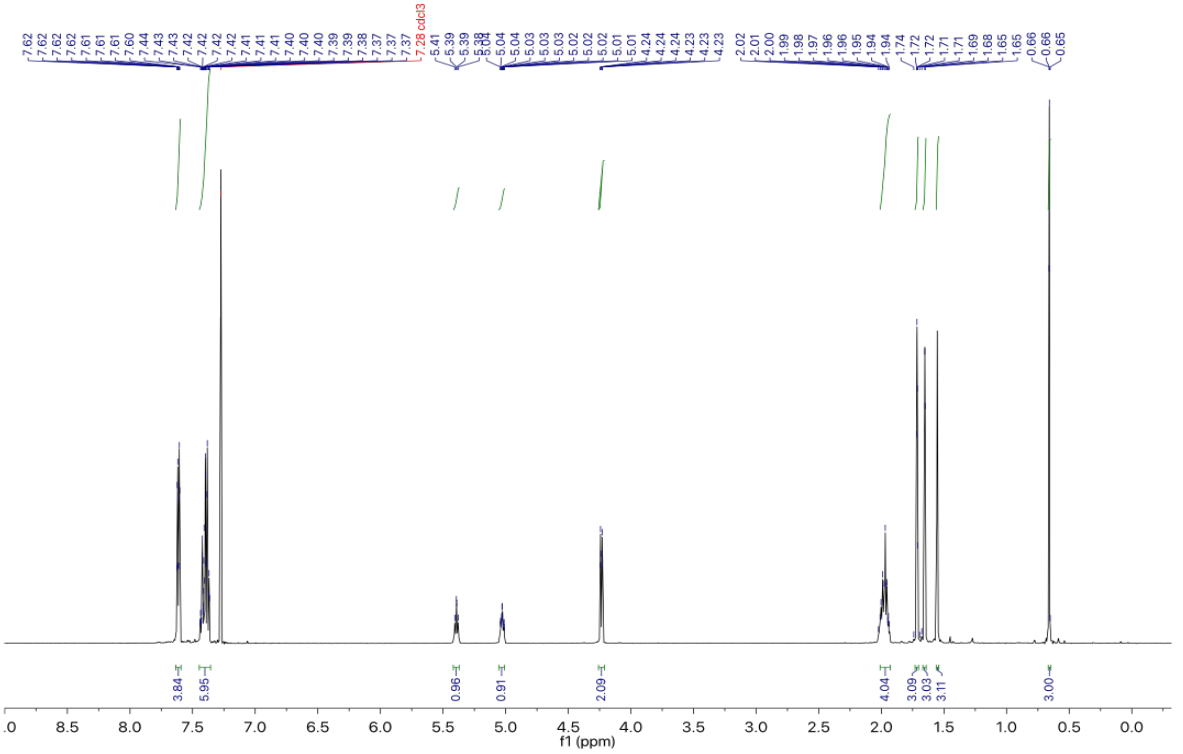
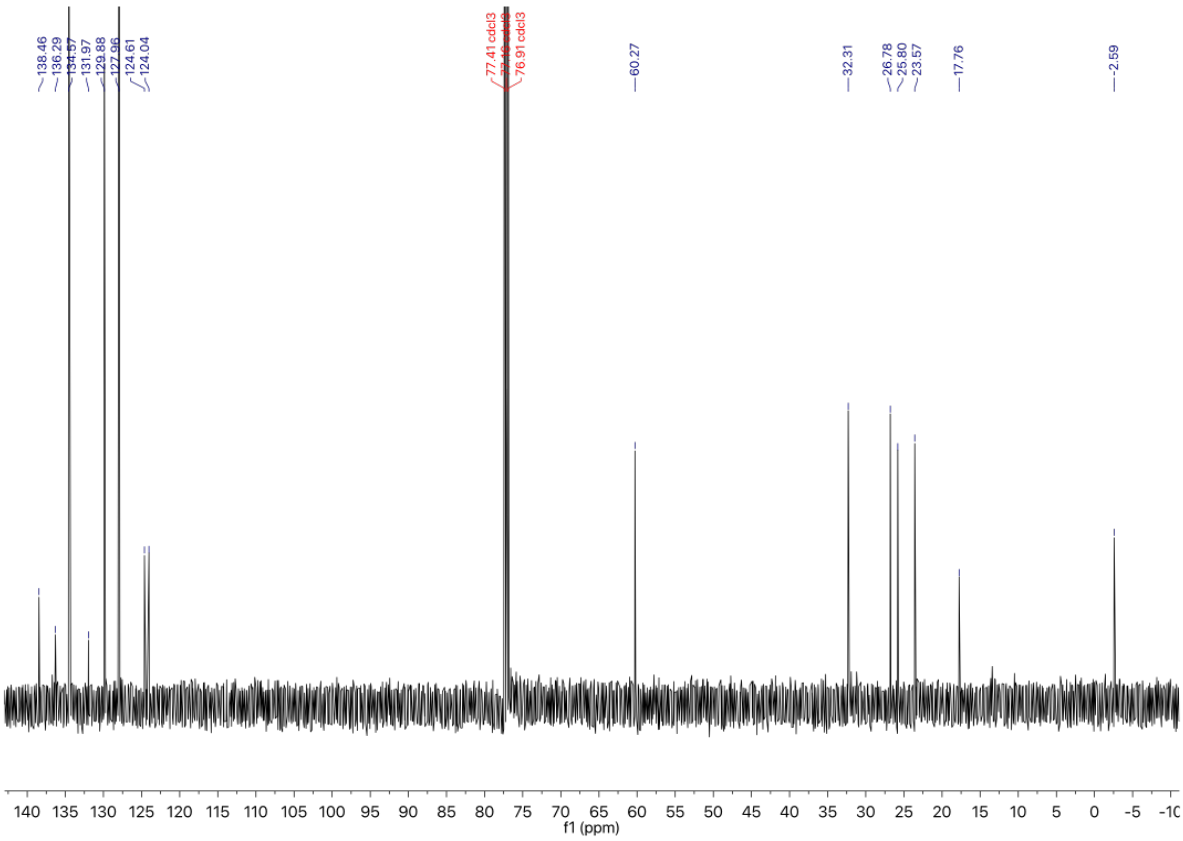
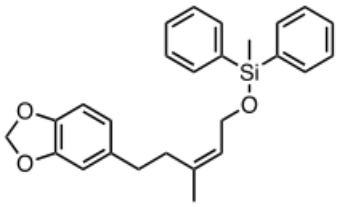
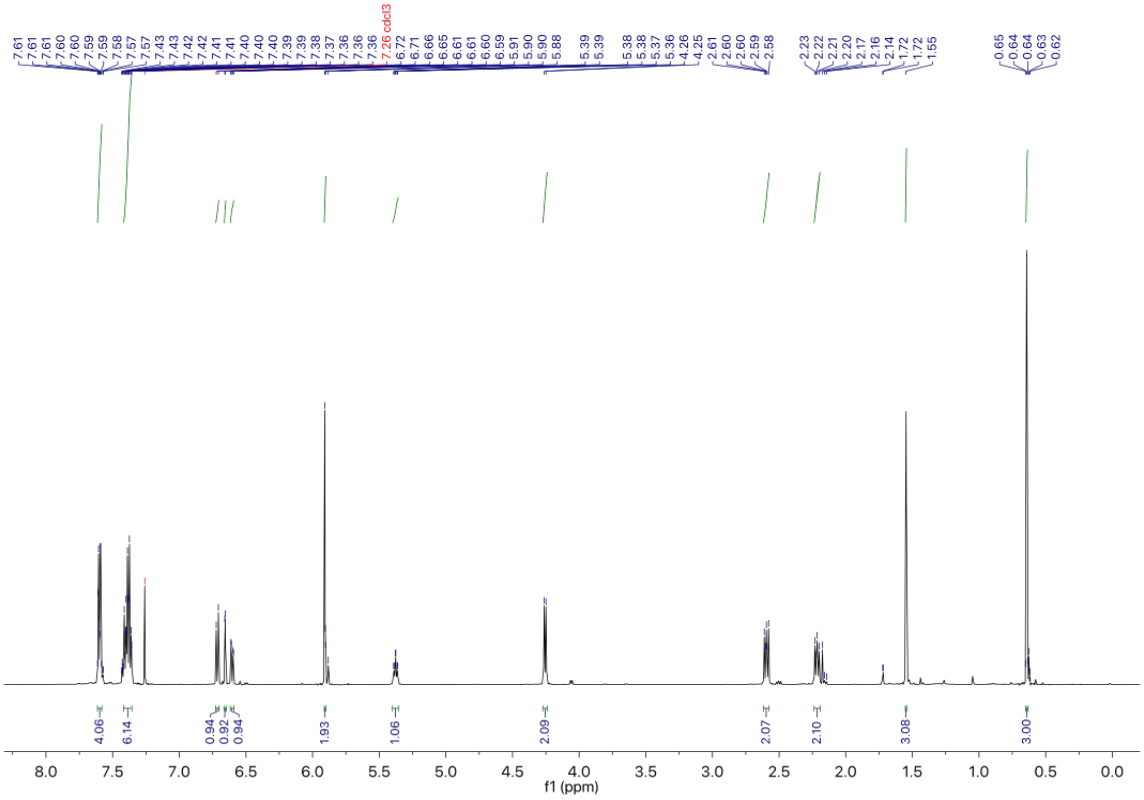
157.86
136.67
136.34
134.49
129.89
127.96
120.54
119.70
77.42 cdcl3
77.16 cdcl3
76.91 cdcl3
63.07
34.90
32.88
24.69
-2.91
f1 (ppm)

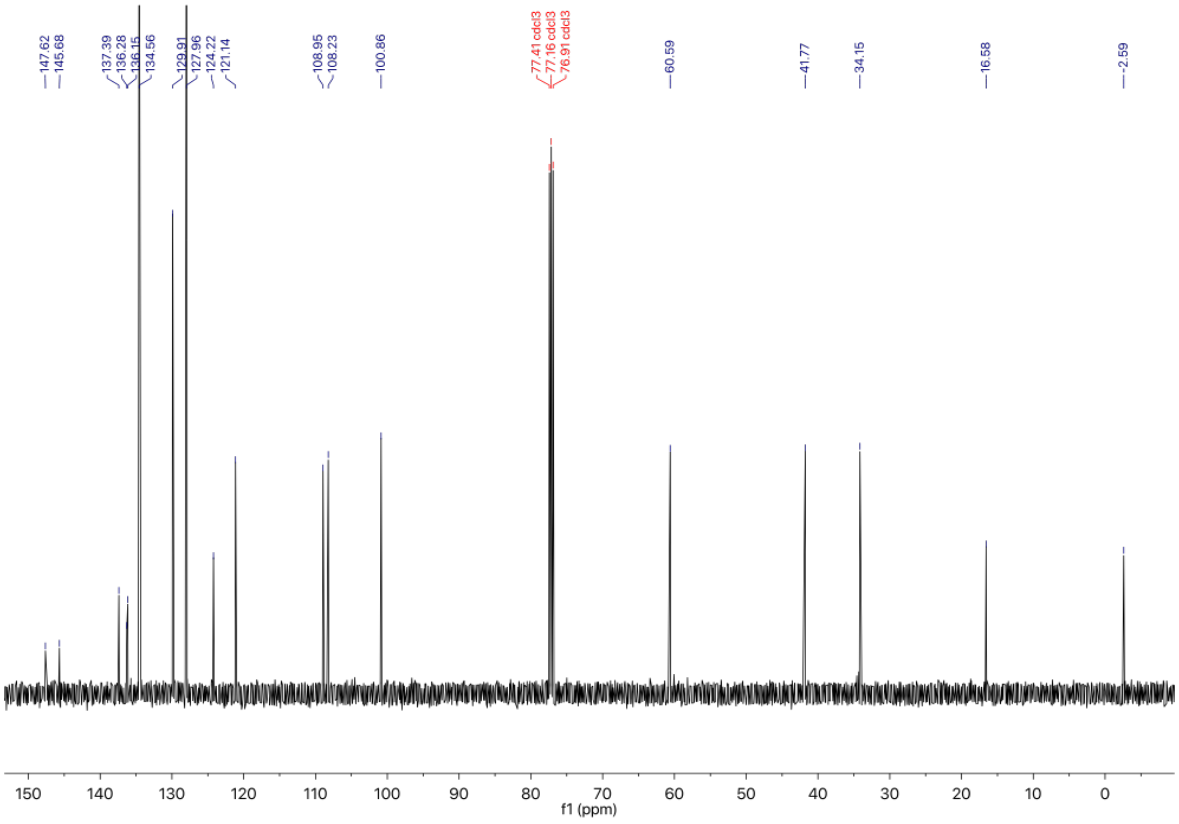


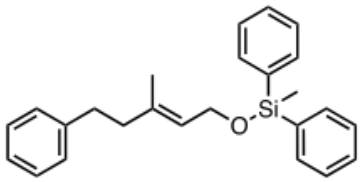

7.62 7.62 7.62 7.62 7.61 7.61 7.61 7.60 7.44 7.43 7.43 7.42 7.42 7.42 7.42 7.41 7.41 7.41 7.40 7.40 7.40 7.39 7.39 7.38 7.37 7.37 7.37 7.28 cdcl3
5.41 5.39 5.39 5.38 5.04 5.04 5.03 5.03 5.03 5.02 5.02 5.02 5.01 5.01 4.24 4.24 4.24 4.23 4.23 4.23
2.02 2.01 2.00 1.99 1.98 1.97 1.96 1.96 1.95 1.94 1.94 1.74 1.72 1.72 1.71 1.71 1.69 1.68 1.65 1.65 0.66 0.66 0.66 0.65
3.84 5.95 0.96 0.91 2.09 4.04 3.09 3.03 3.11 3.00
f1 (ppm)

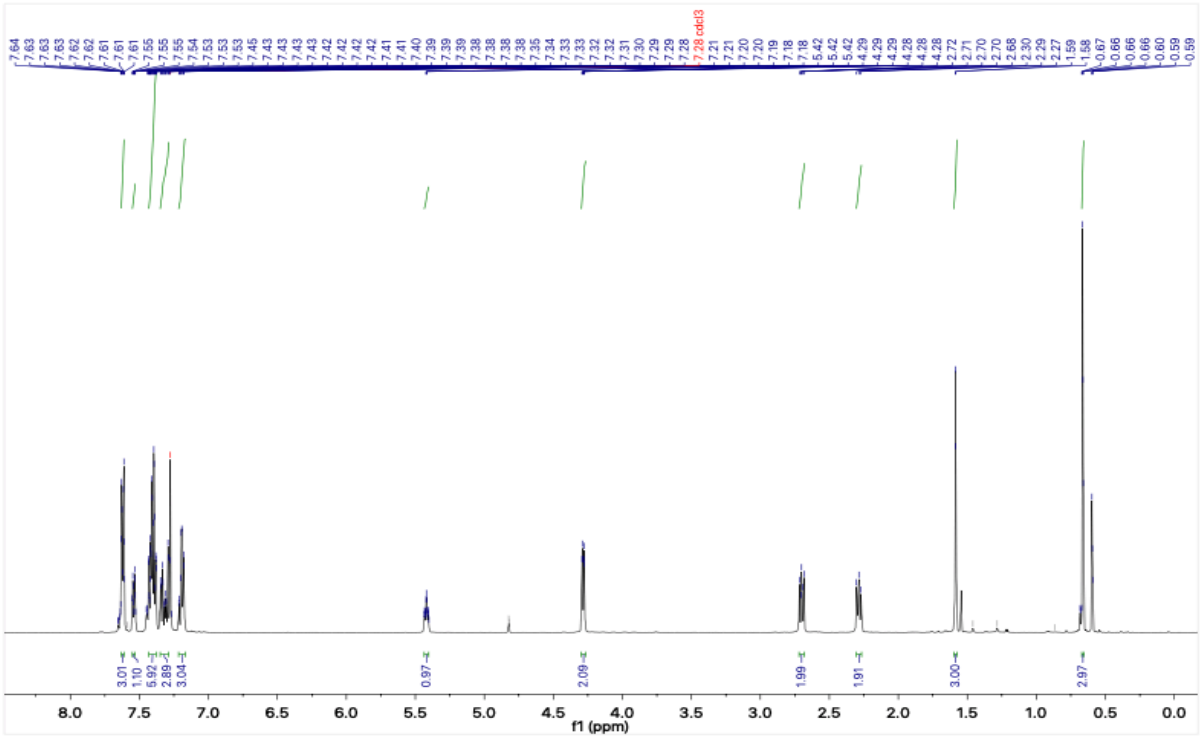

138.46 136.29 134.57 131.97 129.88 127.96 124.61 124.04
77.41 cdcl3 77.16 cdcl3 76.91 cdcl3
60.27
32.31
26.78 25.80 23.57
17.76
-2.59
f1 (ppm)

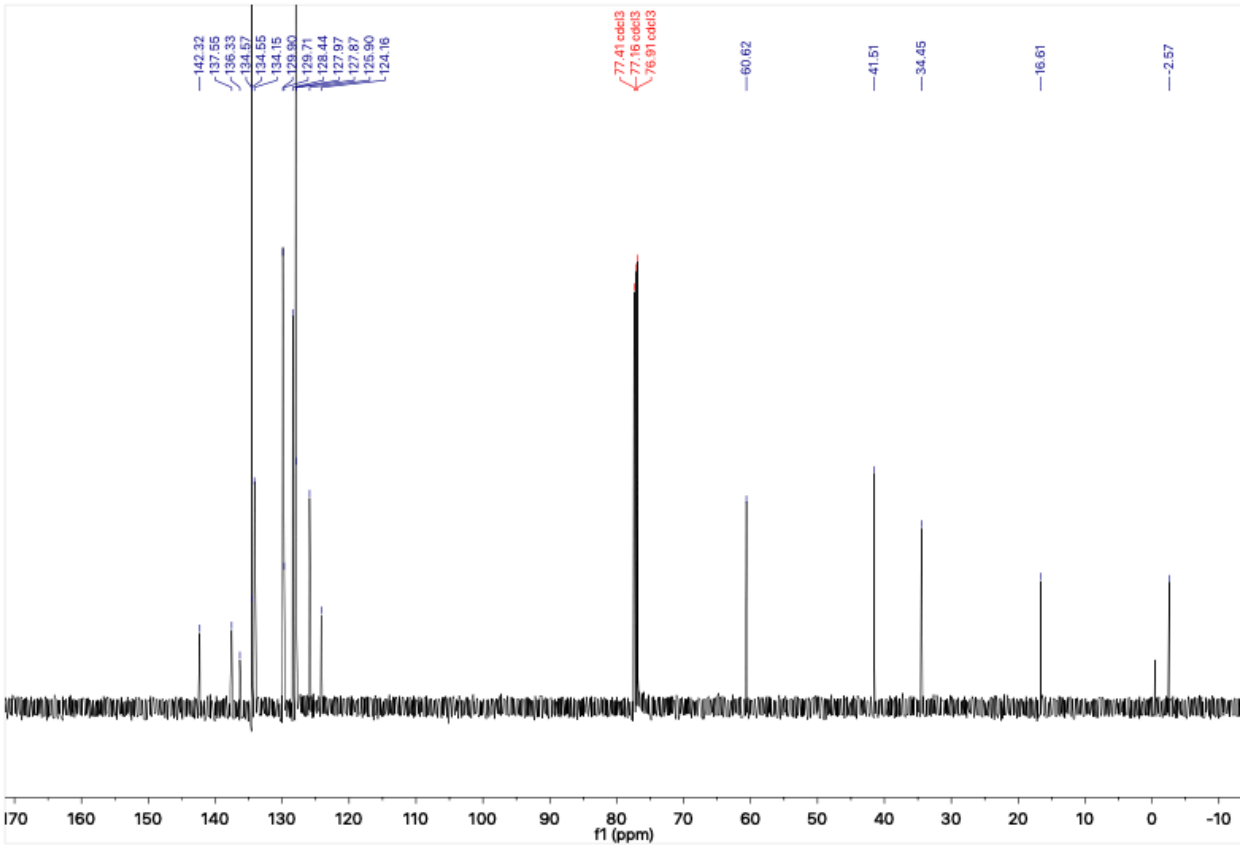


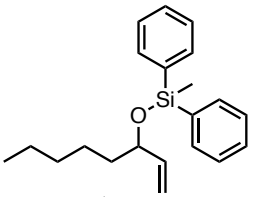

7.61 7.61 7.61 7.60 7.60 7.60 7.59 7.59 7.59 7.58 7.57 7.57 7.43 7.43 7.42 7.42 7.41 7.41 7.40 7.40 7.40 7.39 7.39 7.38 7.37 7.36 7.36 7.36 7.26 cdcl3 6.72 6.71 6.66 6.65 6.61 6.61 6.60 6.59 5.91 5.90 5.90 5.88 5.39 5.39 5.38 5.38 5.37 5.36 4.26 4.25 2.61 2.60 2.60 2.59 2.58 2.23 2.22 2.21 2.20 2.17 2.16 2.14 1.72 1.72 1.55 0.65 0.64 0.64 0.63 0.62
4.06 6.14 0.94 0.92 0.94 1.93 1.06 2.09 2.07 2.10 3.08 3.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

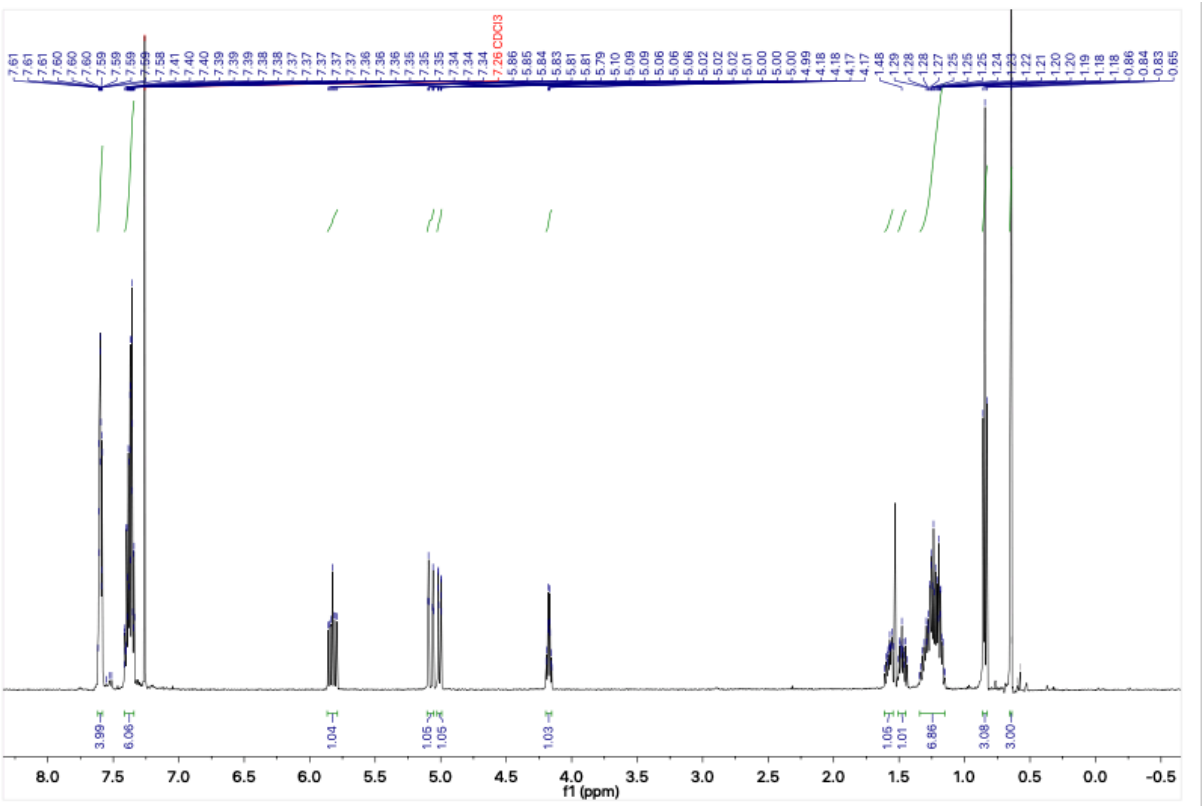

147.62 145.68 137.39 136.28 136.15 134.56 129.91 127.96 124.22 121.14 108.95 108.23 100.86 77.41 cdcl3 77.16 cdcl3 76.91 cdcl3 60.59 41.77 34.15 16.58 -2.59
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)




f1 (ppm)


f1 (ppm)


Si
O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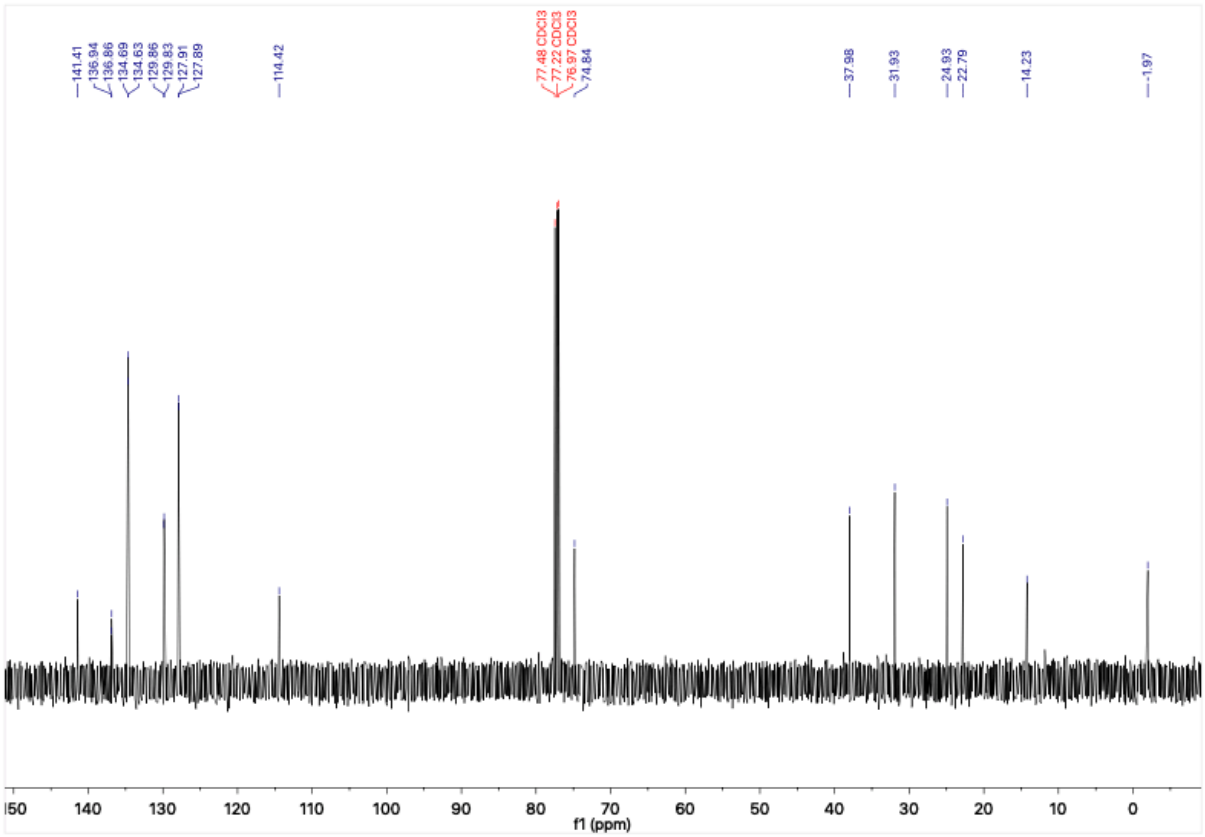
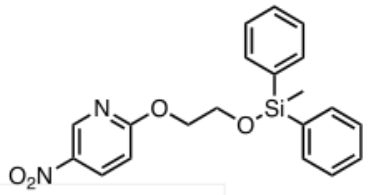
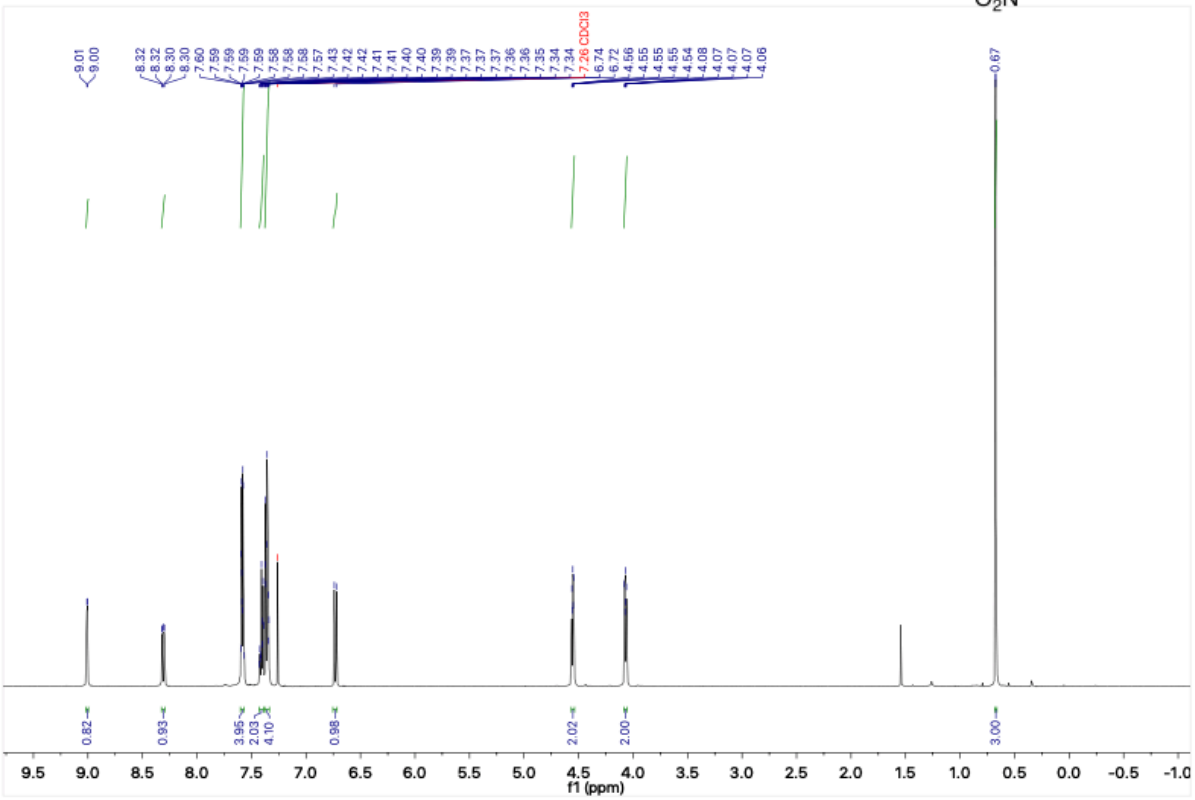
1.05
1.05
1.03
1.05
1.01
6.86
3.08
3.00
f1 (ppm)


141.41
136.94
136.86
134.69
134.63
129.86
129.83
127.91
127.89
114.42
77.48 CDCl3
77.22 CDCl3
76.97 CDCl3
74.84
37.98
31.93
24.93
22.79
14.23
-1.97
f1 (ppm)


N
O
O
Si
O_2N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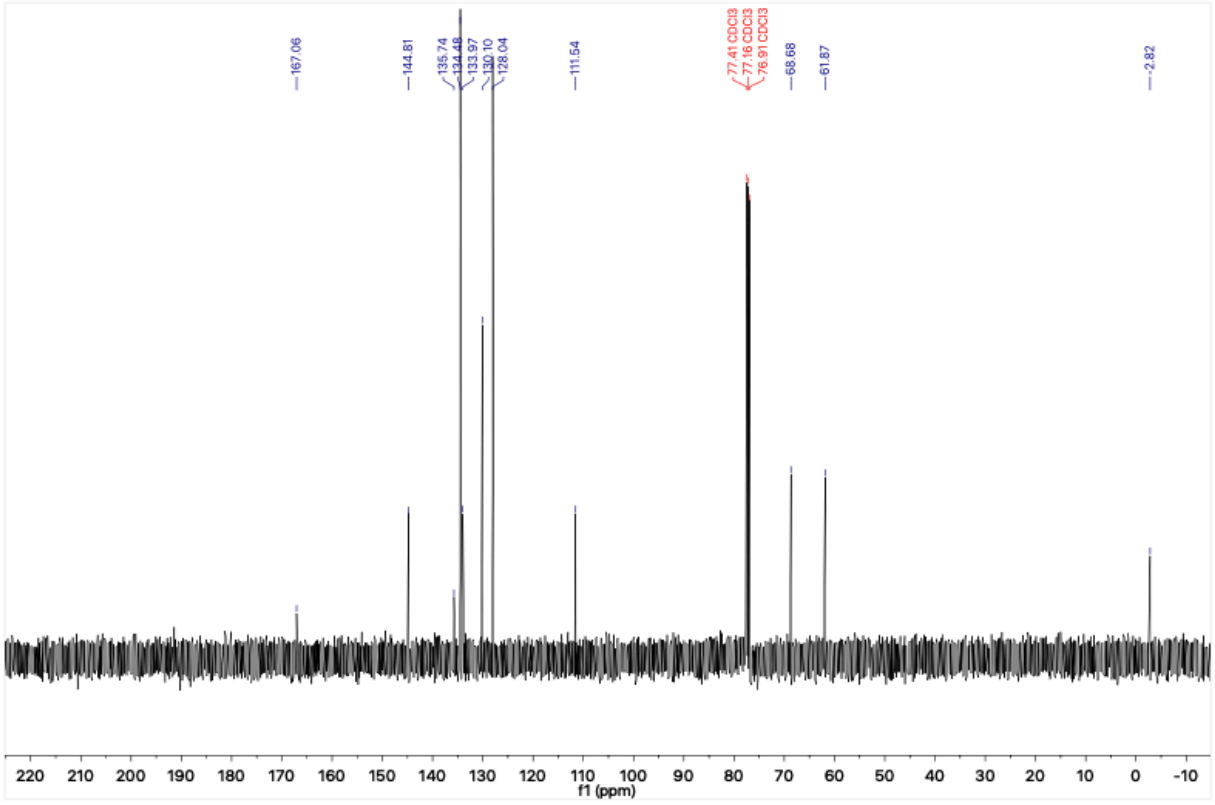
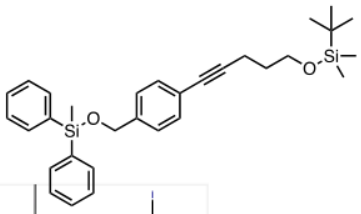
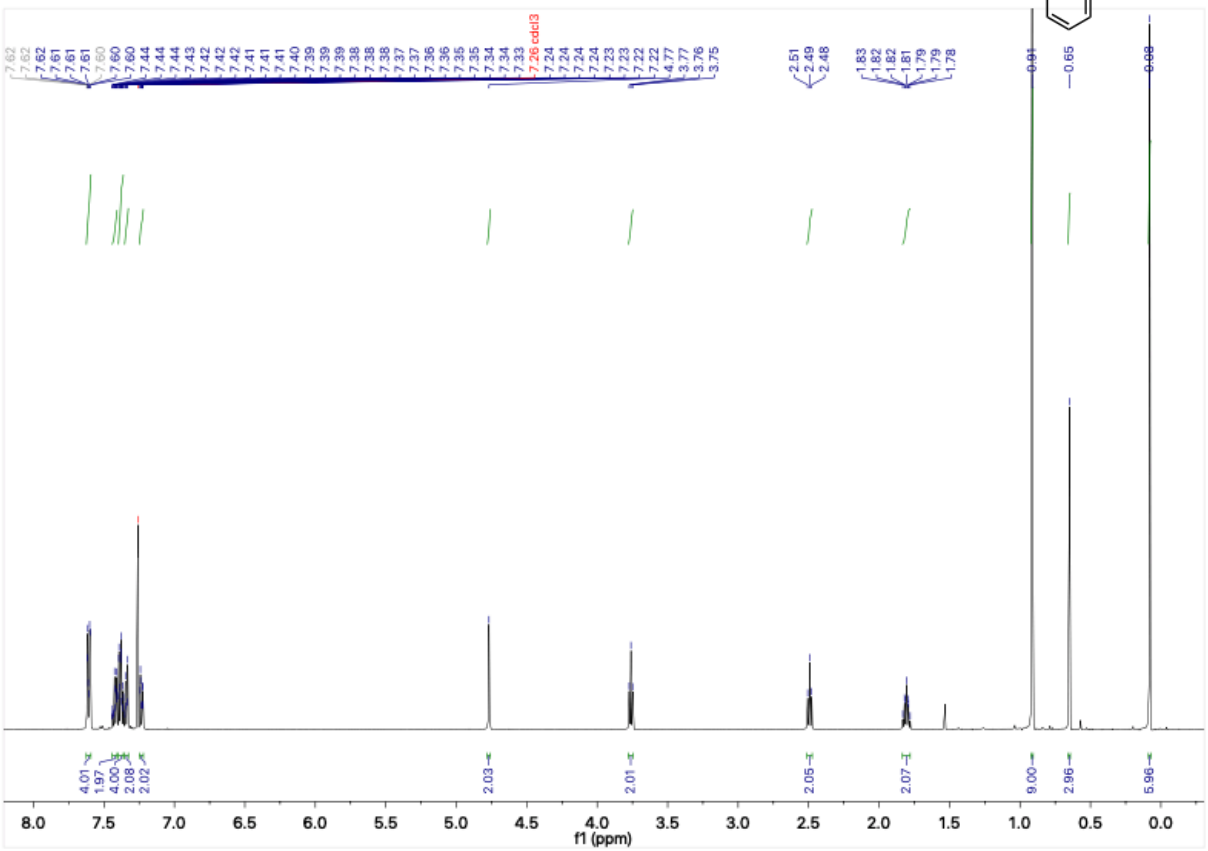
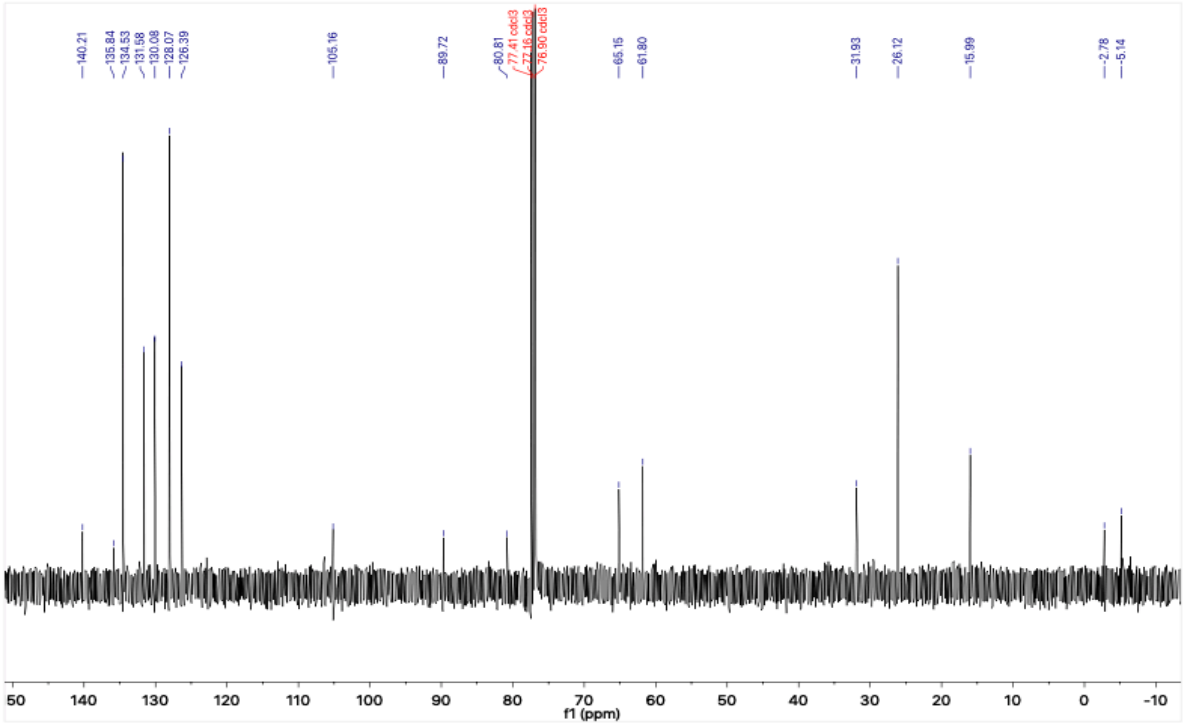
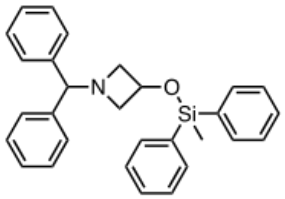
9.00
8.32
8.32
8.30
8.30
7.60
7.59
7.59
7.59
7.59
7.58
7.58
7.58
7.57
7.43
7.42
7.42
7.41
7.41
7.40
7.40
7.39
7.39
7.37
7.37
7.37
7.36
7.36
7.35
7.34
7.34
7.26 CDCl3
6.74
6.72
4.56
4.55
4.55
4.54
4.08
4.07
4.07
4.07
4.06
0.67
0.82
0.93
3.95
2.03
4.10
0.98
2.02
2.00
3.00
f1 (ppm)

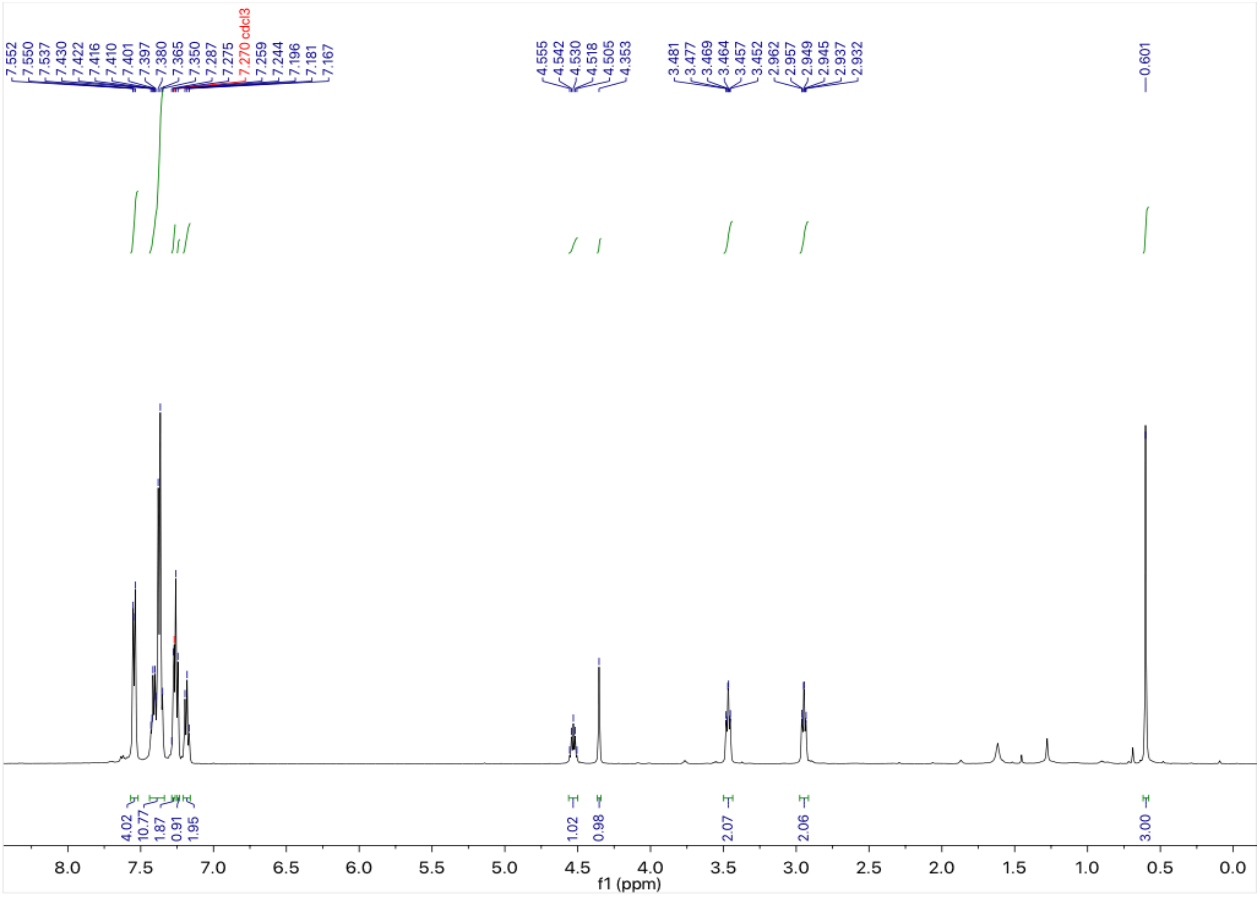

167.06
144.81
135.74
134.48
133.97
130.10
128.04
111.54
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
68.68
61.87
-2.82
f1 (ppm)




7.26 cdcl3
f1 (ppm)


140.21
135.84
134.53
131.58
130.08
128.07
126.39
105.16
89.72
80.81
77.41 cdcl3
77.16 cdcl3
76.90 cdcl3
65.15
61.80
31.93
26.12
15.99
-2.78
-5.14
f1 (ppm)


N
O
Si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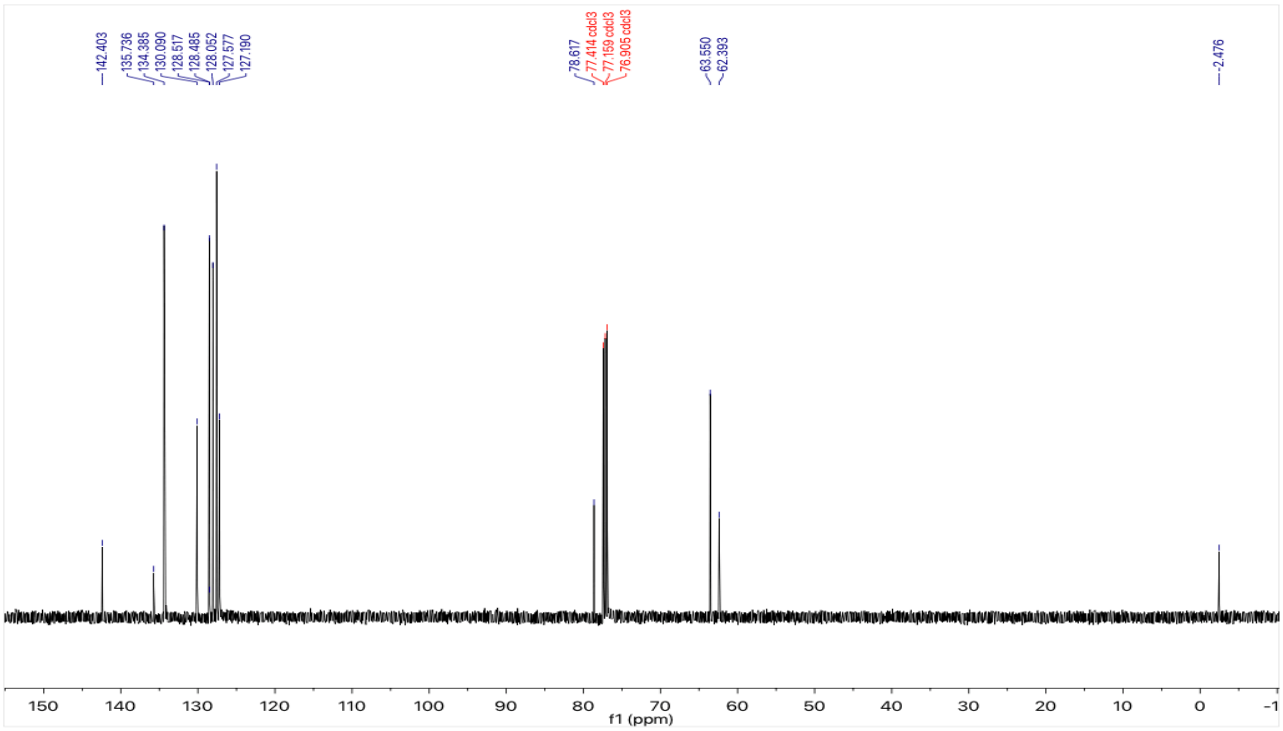
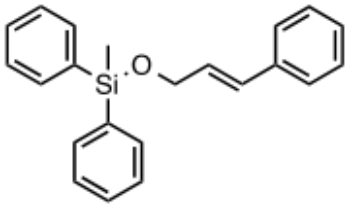
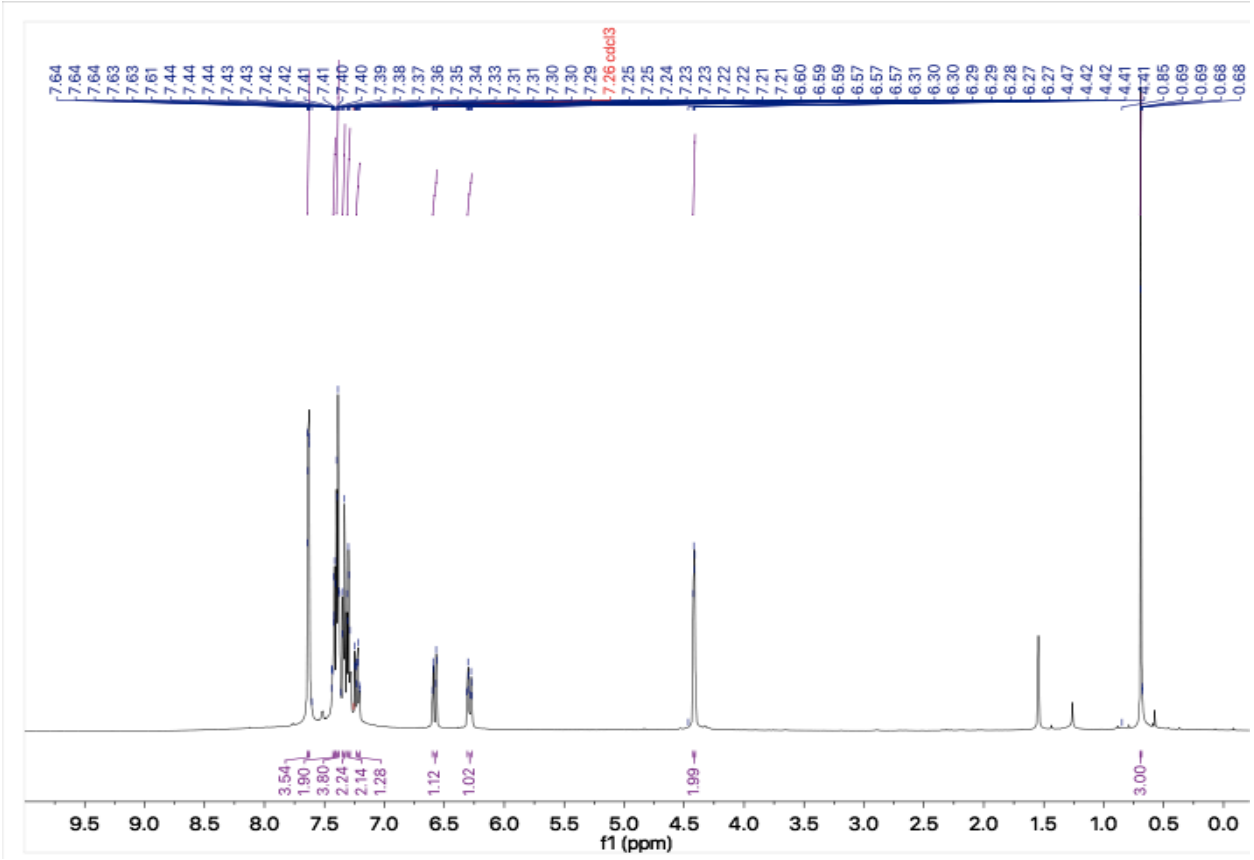
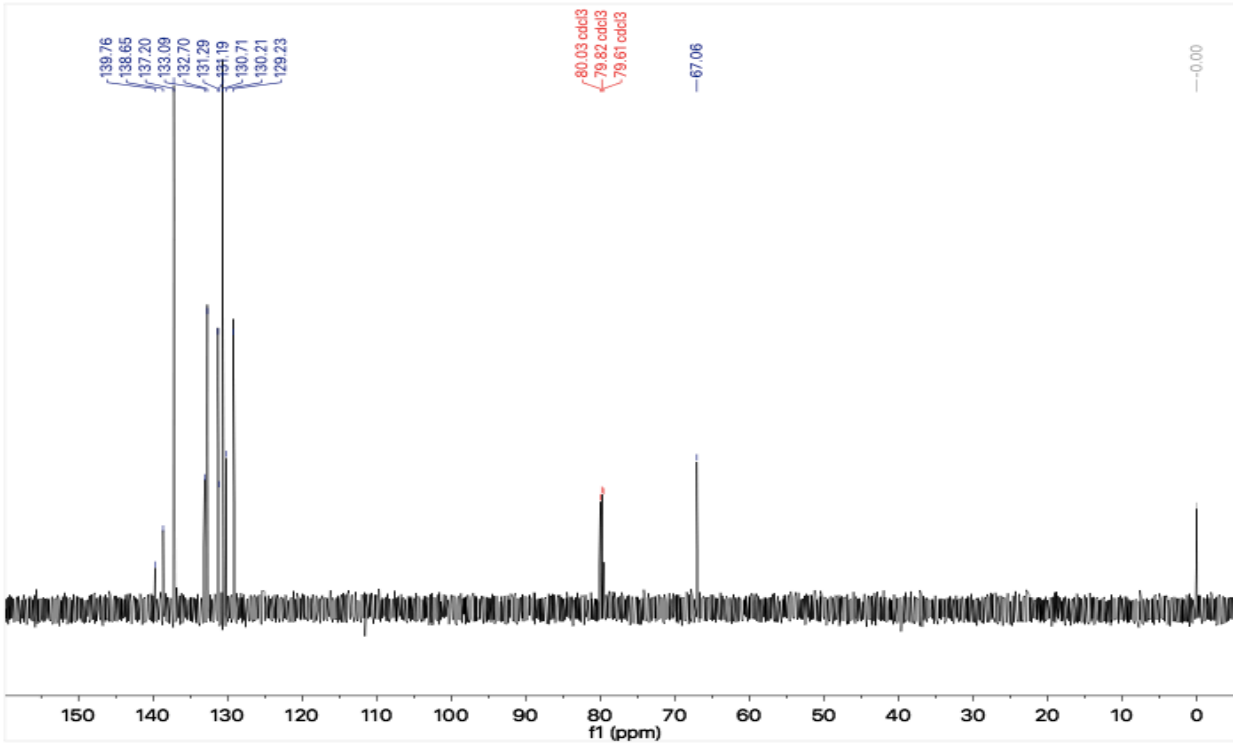
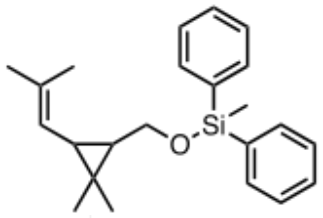
7.550
7.537
7.430
7.422
7.416
7.410
7.401
7.397
7.380
7.365
7.350
7.287
7.275
7.270 cdcl3
7.259
7.244
7.196
7.181
7.167
4.555
4.542
4.530
4.518
4.505
4.353
3.481
3.477
3.469
3.464
3.457
3.452
2.962
2.957
2.949
2.945
2.937
2.932
0.601
4.02
10.77
1.87
0.91
1.95
1.02
0.98
2.07
2.06
3.00
f1 (ppm)

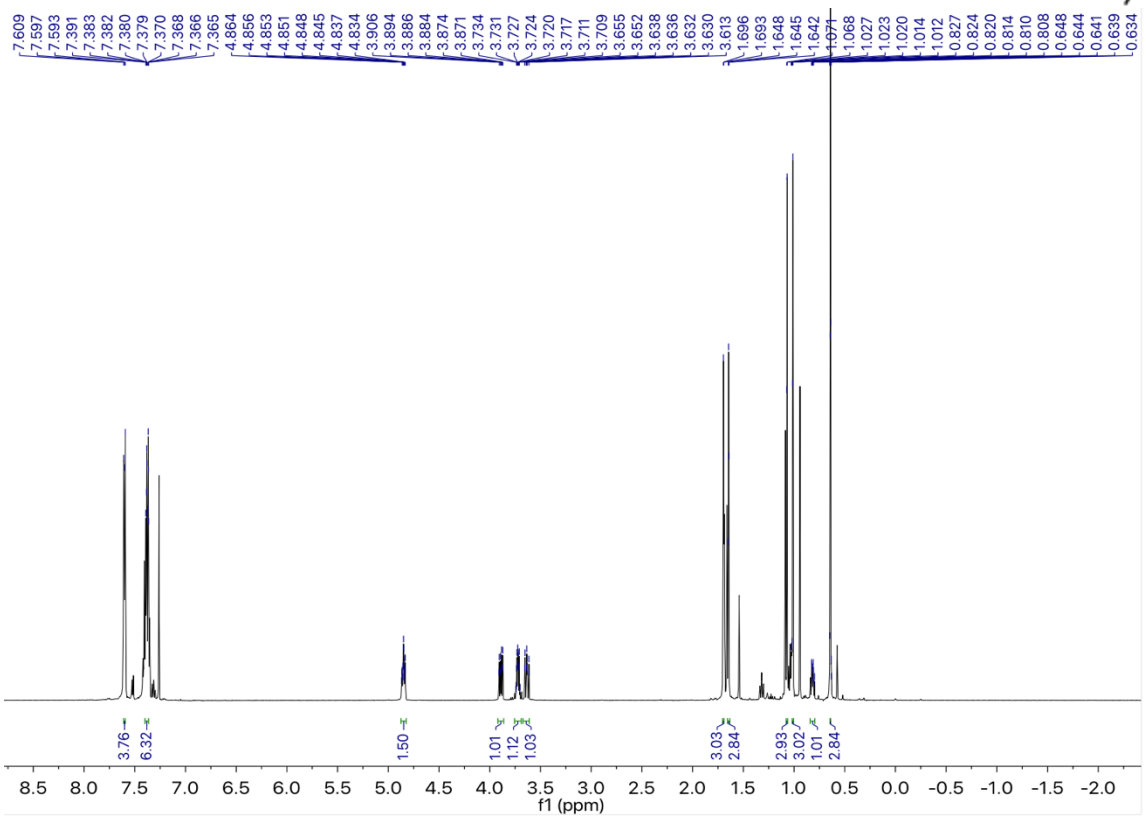

142.403
135.736
134.385
130.090
128.517
128.485
128.052
127.577
127.190
78.617
77.414 cdcl3
77.159 cdcl3
76.905 cdcl3
63.550
62.393
-2.476
f1 (ppm)

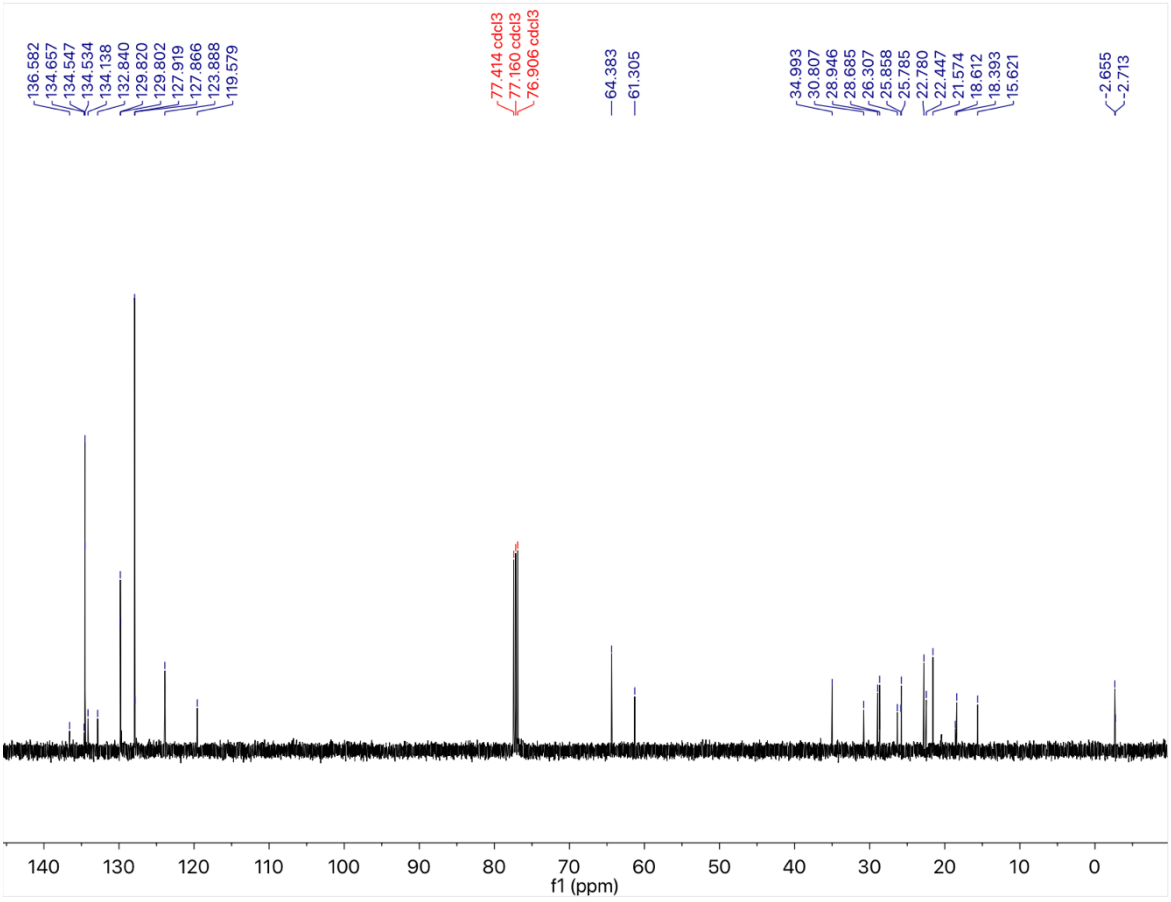

Si
O

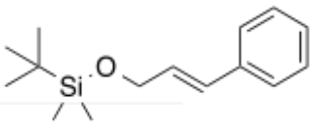

7.64 7.64 7.64 7.63 7.63 7.61 7.44 7.44 7.44 7.43 7.43 7.42 7.42 7.41 7.41 7.40 7.40 7.39 7.38 7.37 7.36 7.35 7.34 7.33 7.31 7.31 7.30 7.30 7.29 7.26 cdcl3 7.25 7.25 7.24 7.23 7.23 7.22 7.22 7.21 7.21 6.60 6.59 6.59 6.57 6.57 6.31 6.30 6.30 6.29 6.29 6.28 6.27 6.27 4.47 4.42 4.42 4.41 4.41 0.85 0.69 0.69 0.68 0.68
3.54 1.90 3.80 2.24 2.14 1.28 1.12 1.02 1.99 3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

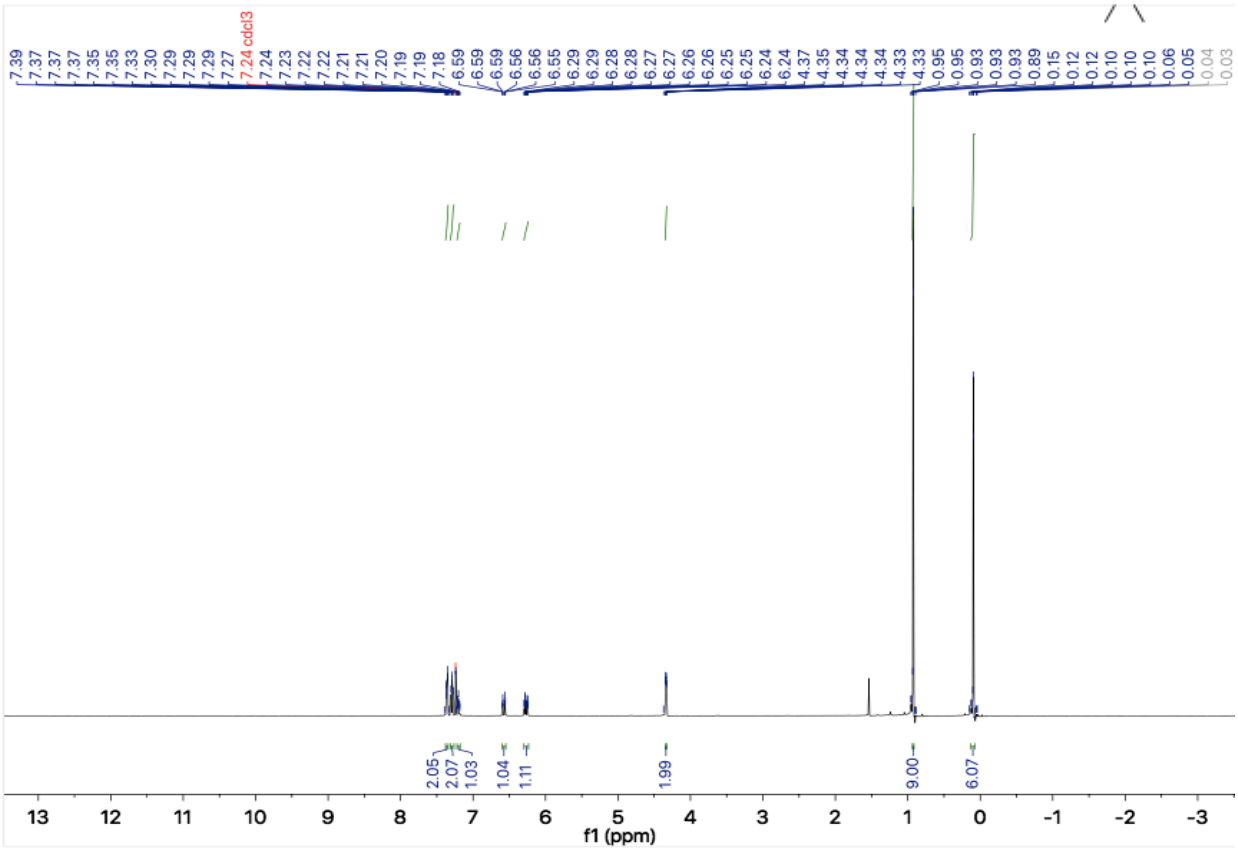

139.76 138.65 137.20 133.09 132.70 131.29 131.19 130.71 130.21 129.23
80.03 cdcl3 79.82 cdcl3 79.61 cdcl3
67.06
0.00
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)




7.609 7.597 7.593 7.391 7.383 7.382 7.380 7.379 7.370 7.368 7.366 7.365
4.864 4.856 4.853 4.851 4.848 4.845 4.837 4.834 3.906 3.894 3.886 3.884 3.874 3.871 3.734 3.731 3.727 3.724 3.720 3.717 3.711 3.709 3.655 3.652 3.638 3.636 3.632 3.630 3.613 1.696 1.693 1.648 1.645 1.642 1.071 1.068 1.027 1.023 1.020 1.014 1.012 0.827 0.824 0.820 0.814 0.810 0.808 0.648 0.644 0.641 0.639 0.634
3.76 6.32 1.50 1.01 1.12 1.03 3.03 2.84 2.93 3.02 1.01 2.84
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)


136.582 134.657 134.547 134.534 134.138 132.840 129.820 129.802 127.919 127.866 123.888 119.579
77.414 cdcl3 77.160 cdcl3 76.906 cdcl3
64.383 61.305
34.993 30.807 28.946 28.685 26.307 25.858 25.785 22.780 22.447 21.574 18.612 18.393 15.621
-2.655 -2.713
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Si
O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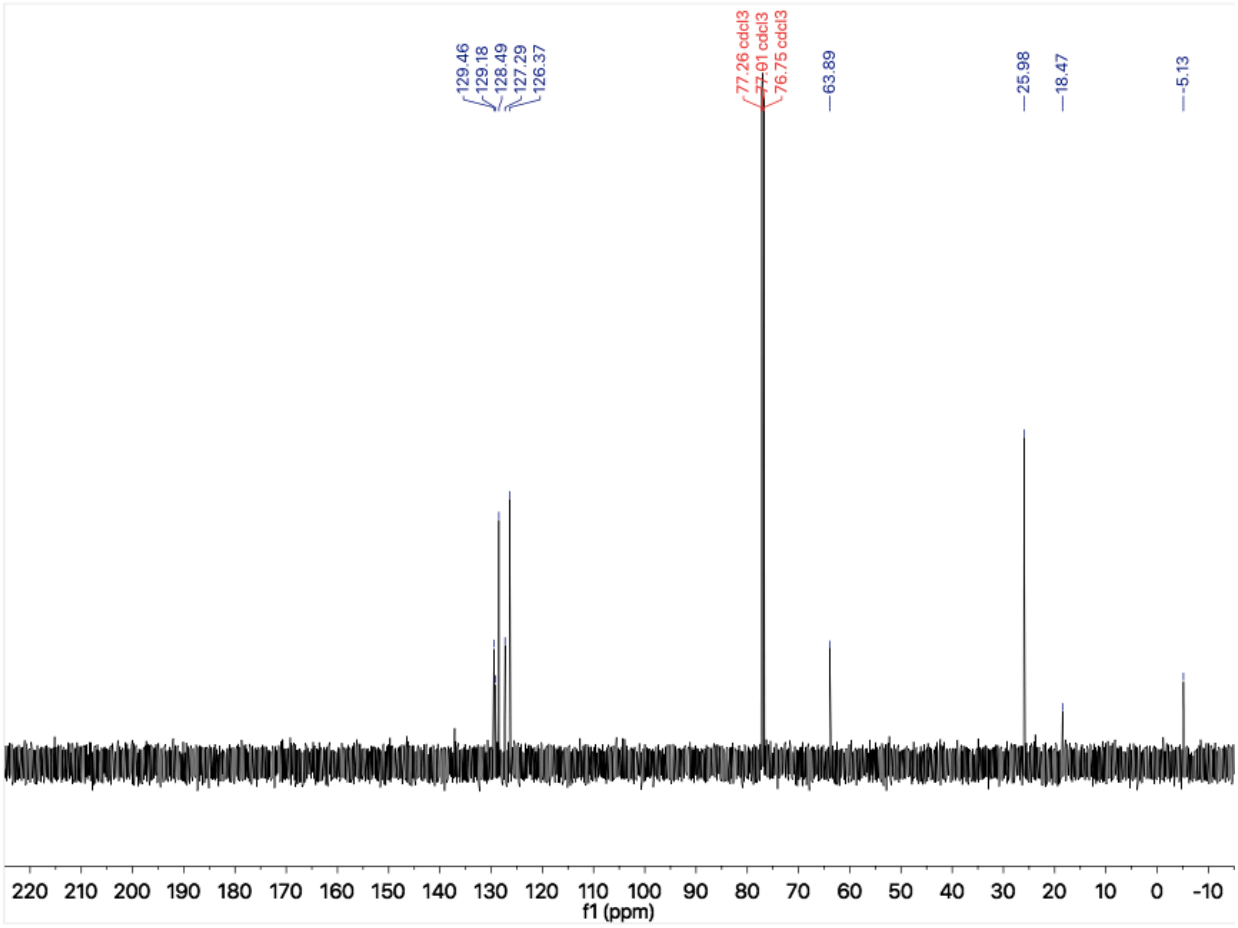
2.07
1.03
1.04
1.11
1.99
9.00
6.07
f1 (ppm)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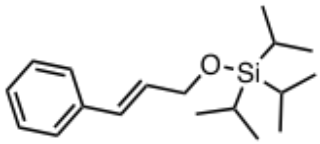
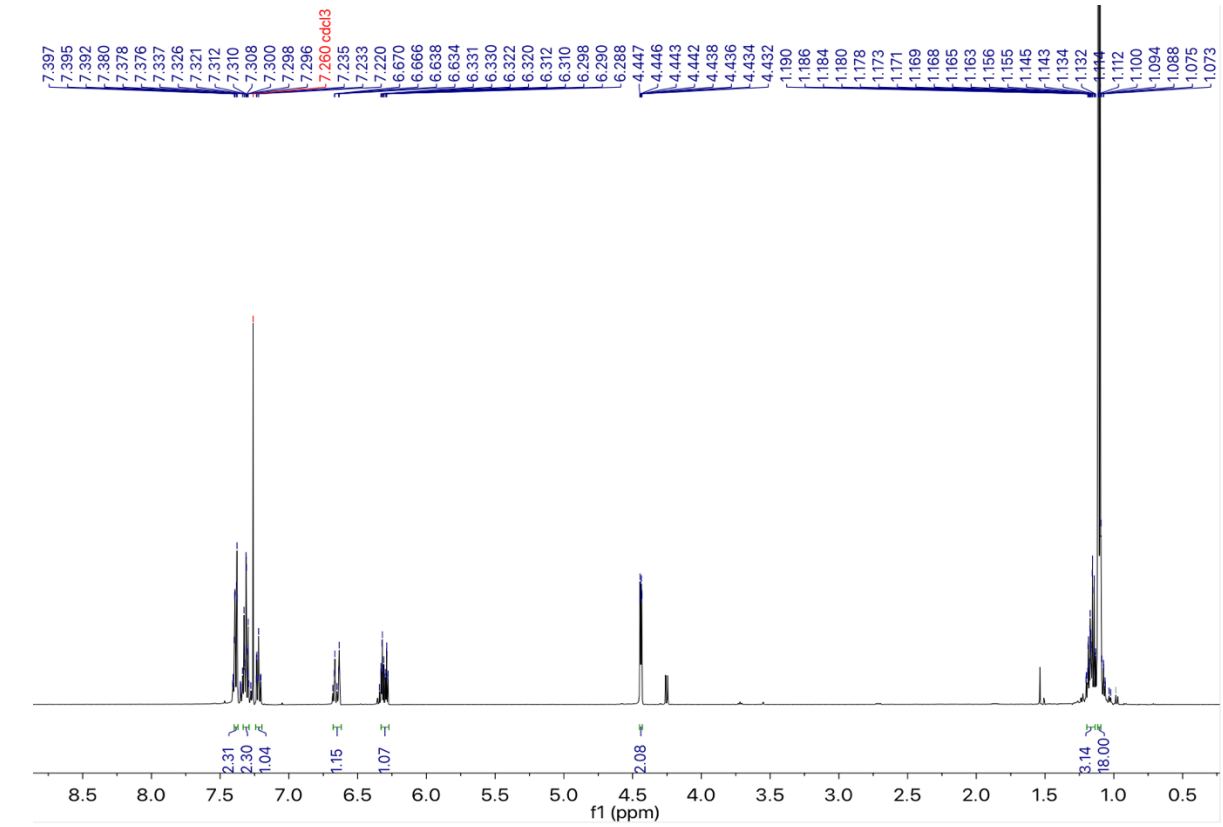
129.18
128.49
127.29
126.37
77.26 cdcl3
77.01 cdcl3
76.75 cdcl3
63.89
25.98
18.47
-5.13
f1 (ppm)

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7.337
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7.298
7.296
7.260 cdcl3
7.235
7.233
7.220
6.670
6.666
6.638
6.634
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6.330
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6.320
6.312
6.310
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1.173
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1.143
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1.132
1.114
1.112
1.100
1.094
1.088
1.075
1.073
2.31
2.30
1.04
1.15
1.07
2.08
3.14
18.00
8.5
8.0
7.5
7.0
6.5
6.0
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4.0
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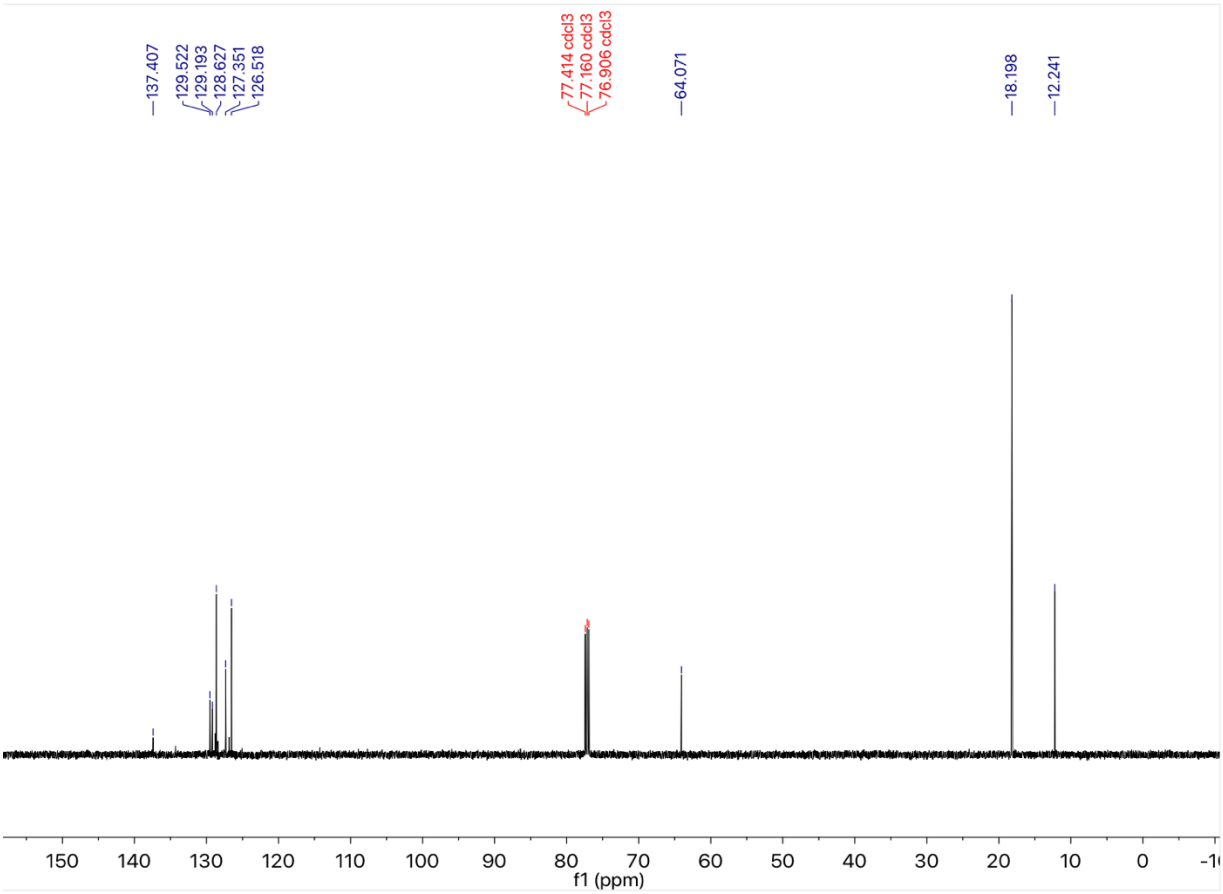
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


137.407
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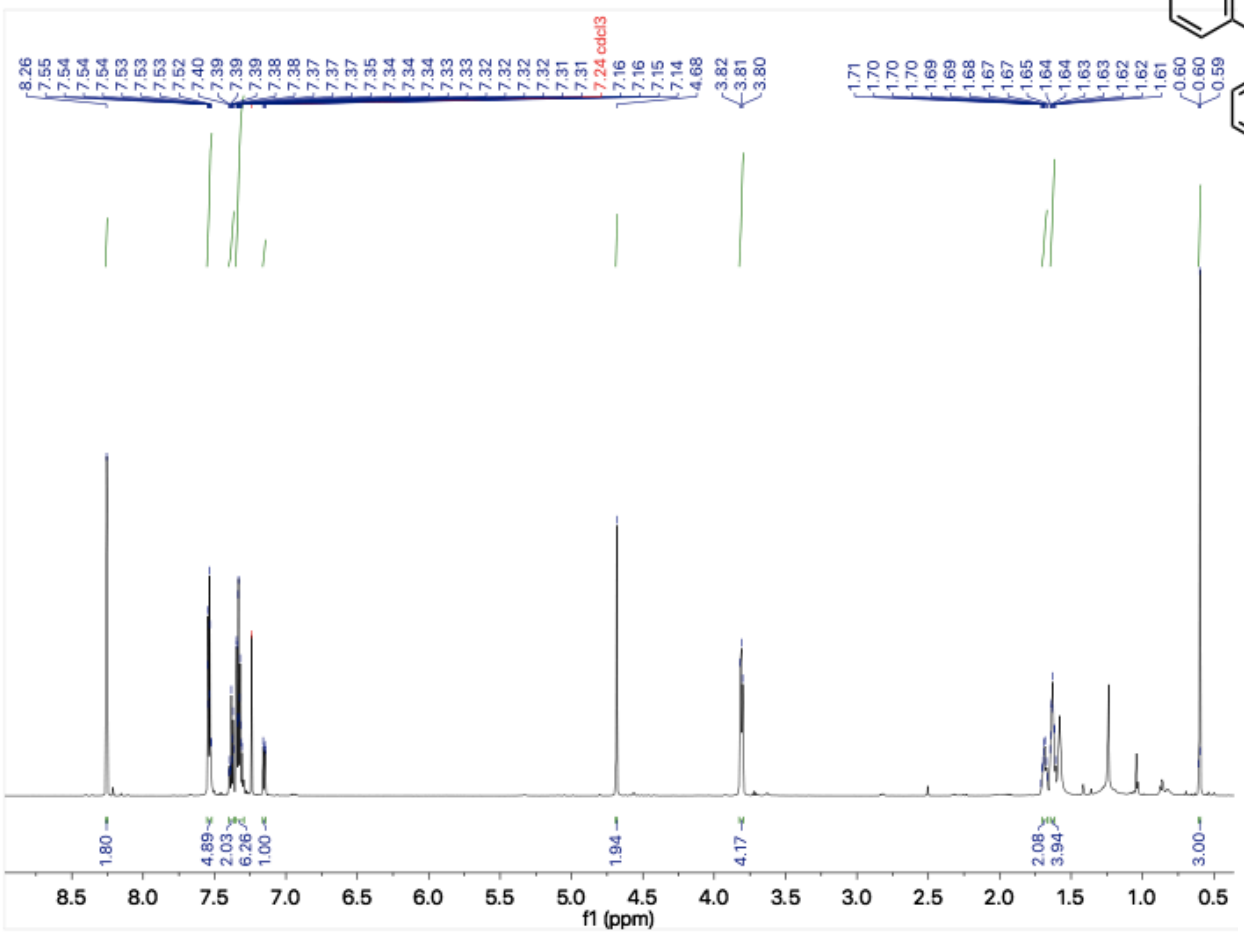
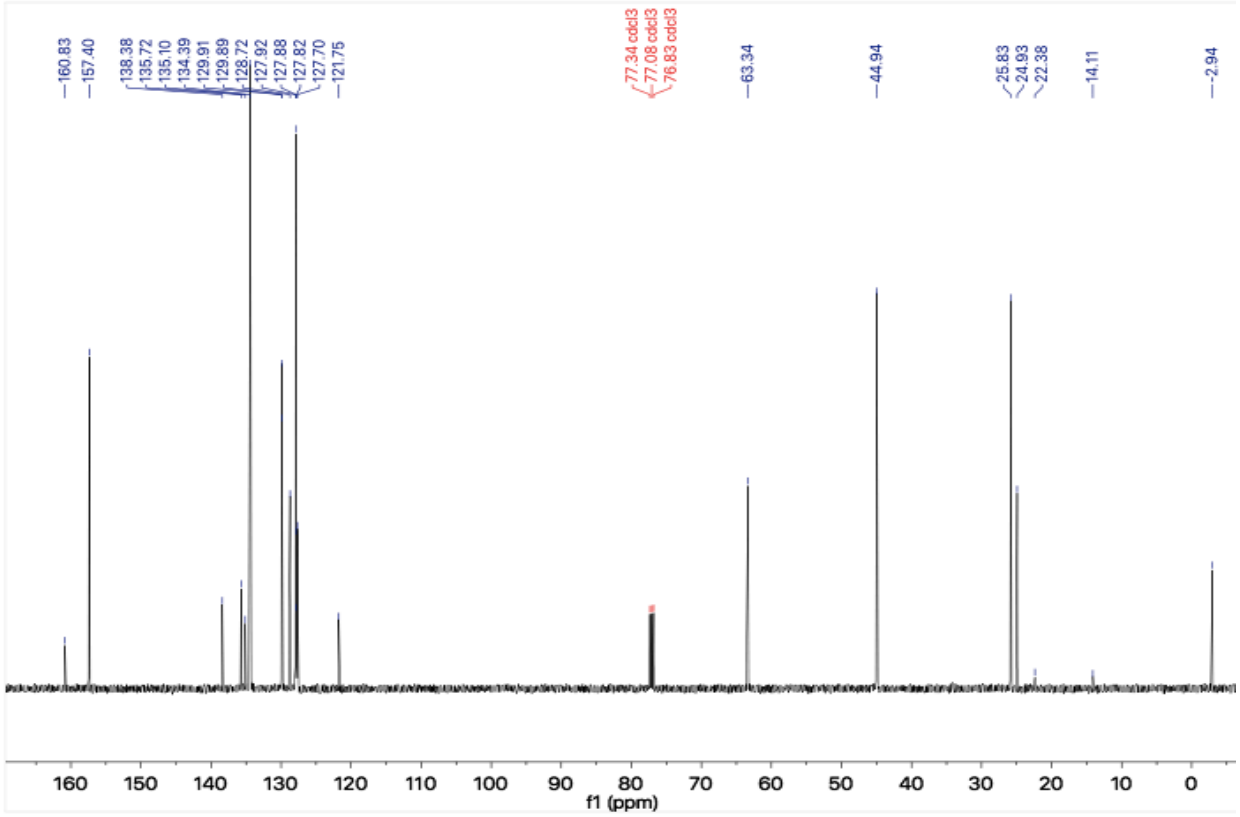
128.627
127.351
126.518
77.414 cdcl3
77.160 cdcl3
76.906 cdcl3
64.071
18.198
12.241
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.26 7.55 7.54 7.54 7.54 7.53 7.53 7.53 7.52 7.40 7.39 7.39 7.39 7.38 7.38 7.37 7.37 7.37 7.35 7.34 7.34 7.34 7.33 7.33 7.32 7.32 7.32 7.32 7.31 7.31 7.24 cdcl3 7.16 7.16 7.15 7.14 4.68 3.82 3.81 3.80
1.71 1.70 1.70 1.70 1.69 1.69 1.68 1.67 1.67 1.65 1.64 1.64 1.63 1.63 1.62 1.62 1.61 0.60 0.60 0.59
1.80 4.89 2.03 6.26 1.00 1.94 4.17 2.08 3.94 3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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77.34 cdcl3 77.08 cdcl3 76.83 cdcl3
63.34 44.94 25.83 24.93 22.38 14.11 2.94
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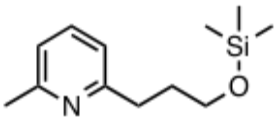
f1 (ppm)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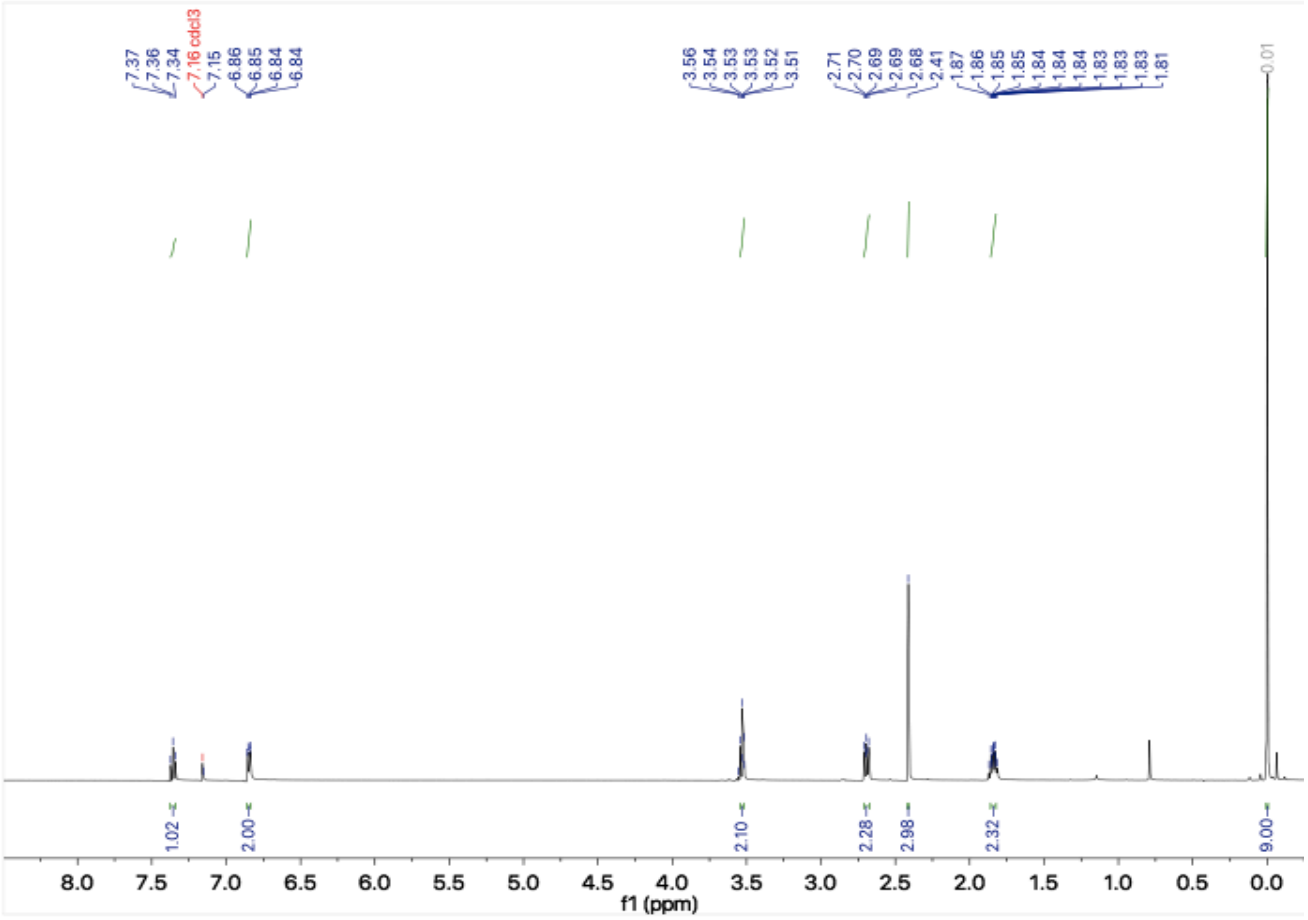
O
N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6.84
3.56
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3.53
3.53
3.52
3.51
2.71
2.70
2.69
2.69
2.68
2.41
1.87
1.86
1.85
1.85
1.84
1.84
1.84
1.83
1.83
1.83
1.81
0.01
1.02
2.00
2.10
2.28
2.98
2.32
9.00
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
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1.0
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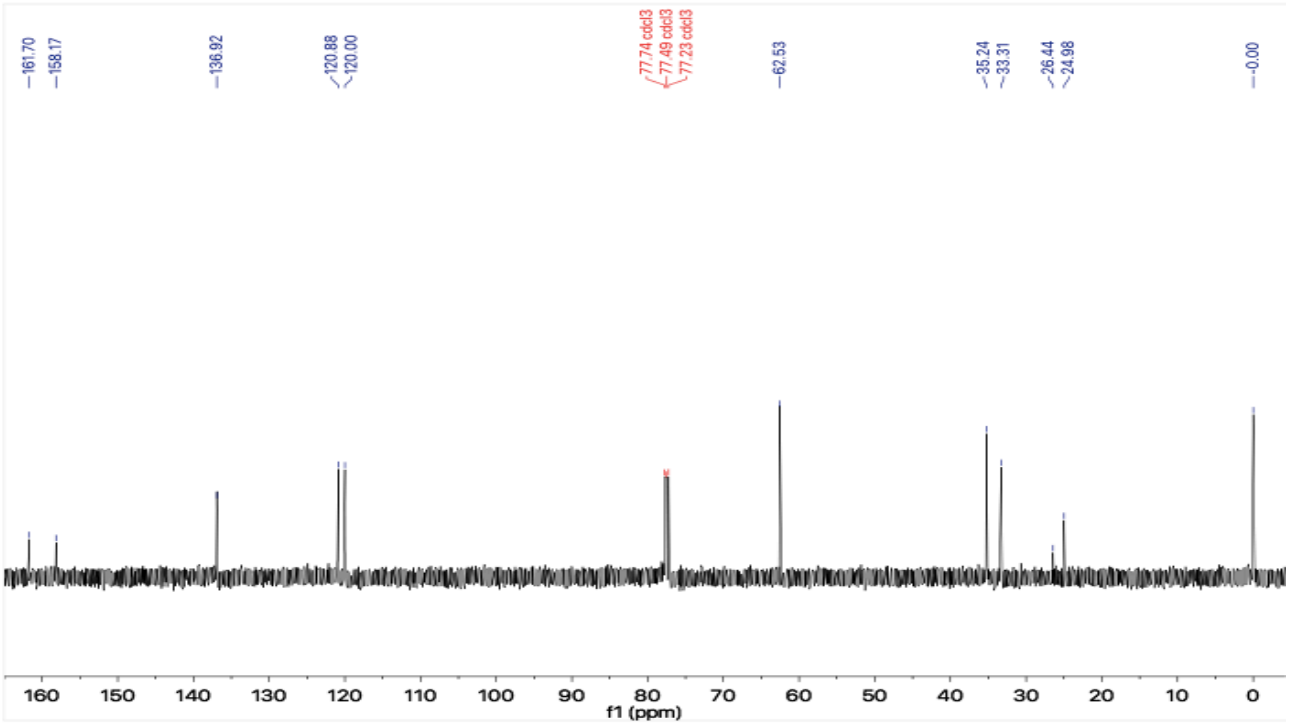
0.0
f1 (ppm)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62.53
35.24
33.31
26.44
24.98
-0.00
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130
120
110
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90
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50
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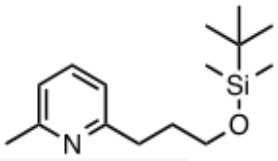
f1 (ppm)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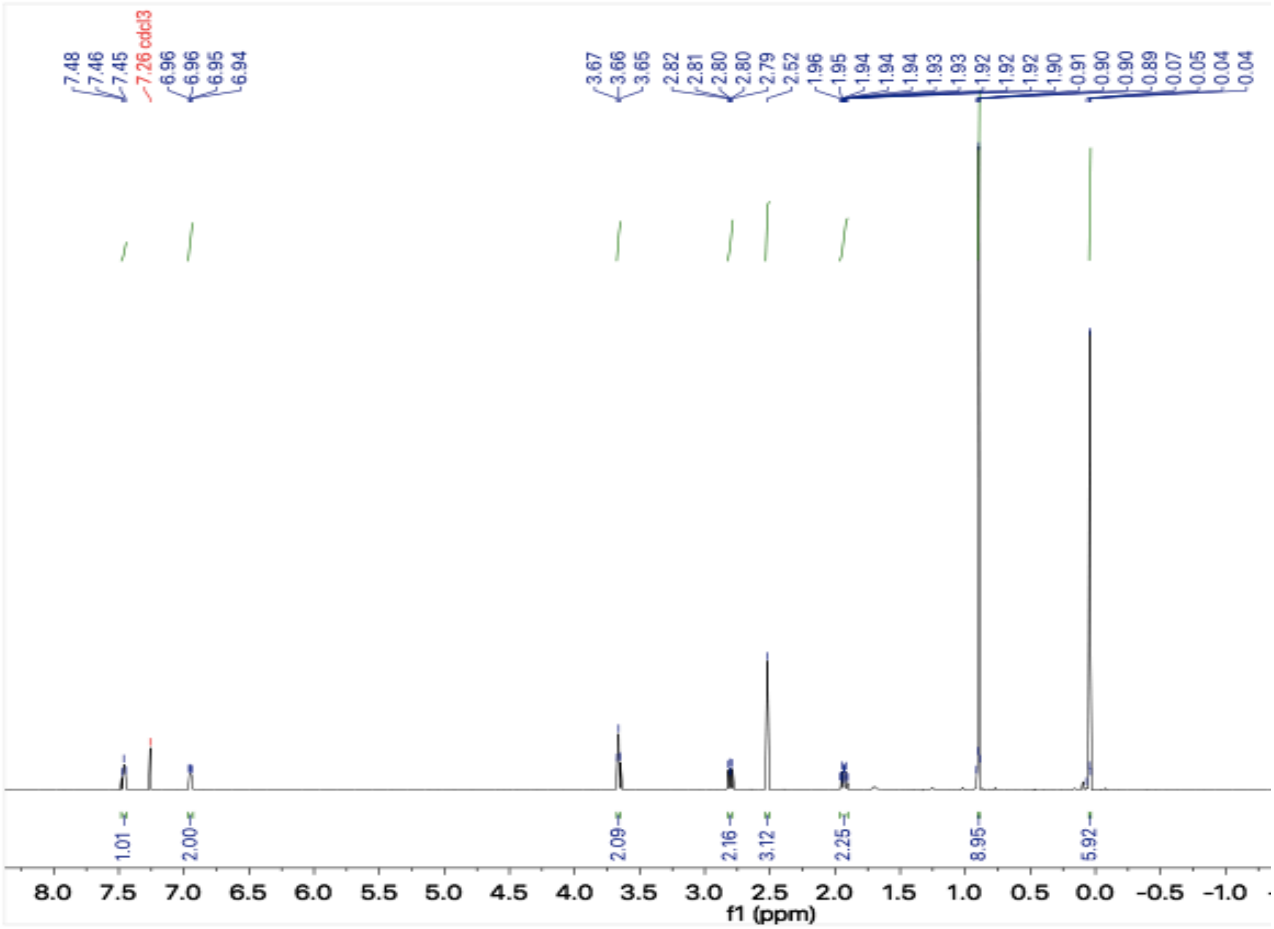
N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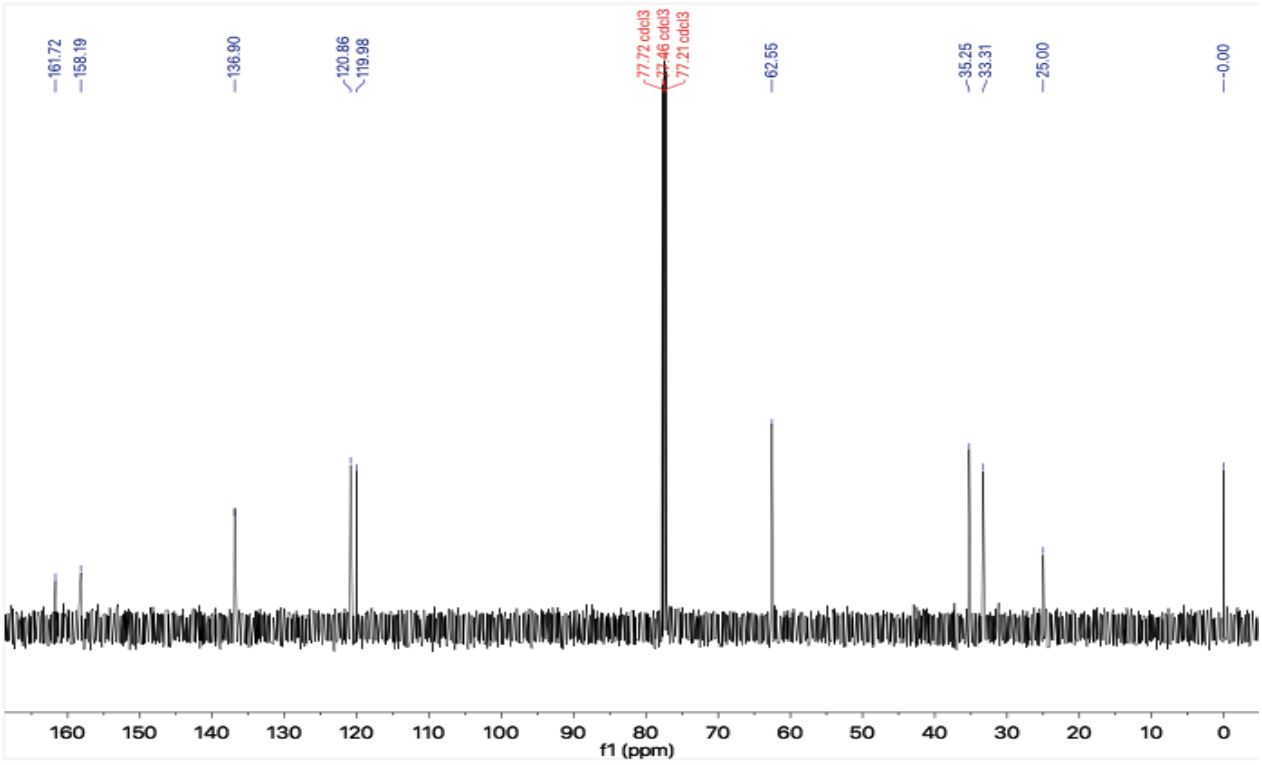
7.45
7.26 cdcl3
6.96
6.96
6.95
6.94
3.67
3.66
3.65
2.82
2.81
2.80
2.80
2.79
2.52
1.96
1.95
1.94
1.94
1.94
1.93
1.93
1.92
1.92
1.92
1.90
0.91
0.90
0.90
0.89
0.07
0.05
0.04
0.04
1.01
2.00
2.09
2.16
3.12
2.25
8.95
5.92
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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120.86
119.98
77.72 cdcl3
77.46 cdcl3
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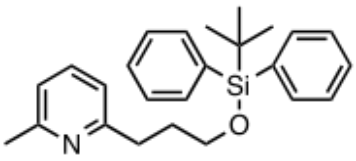
62.55
35.25
33.31
25.00
0.00
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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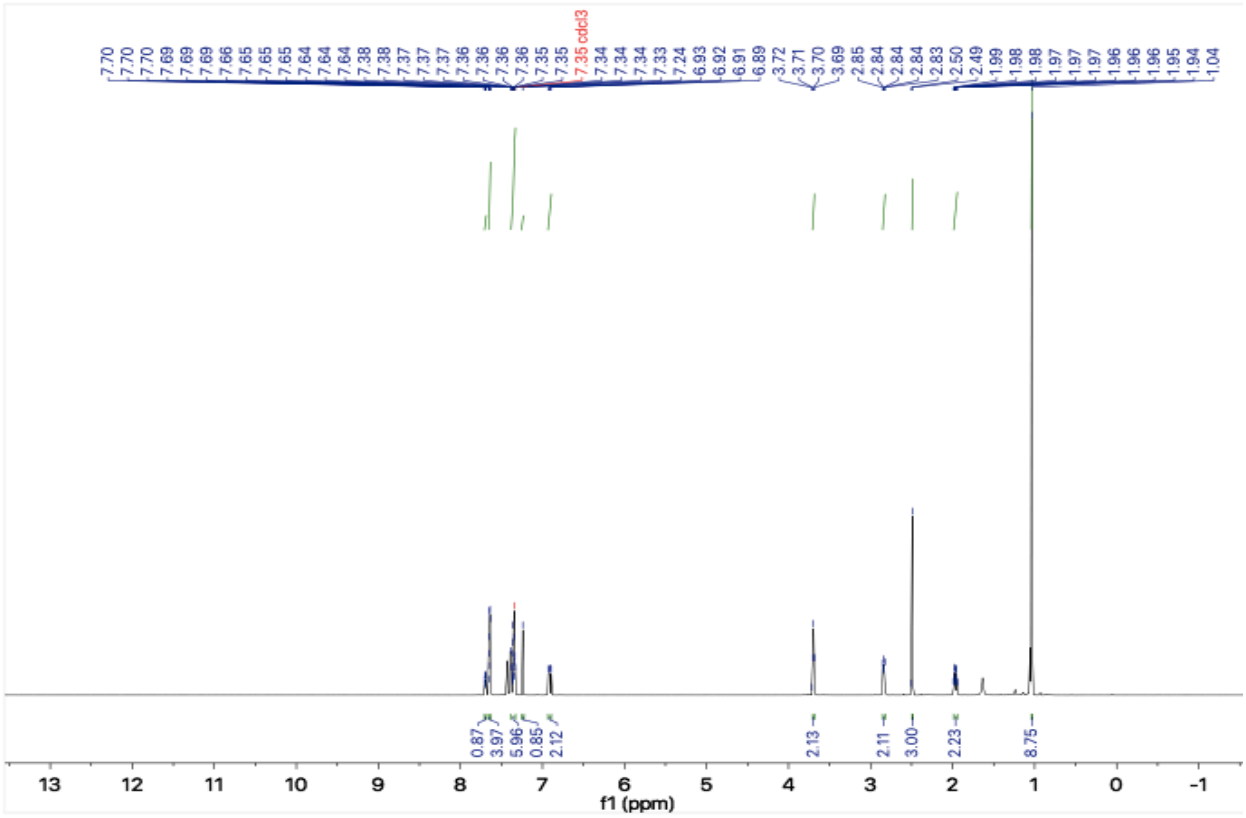
N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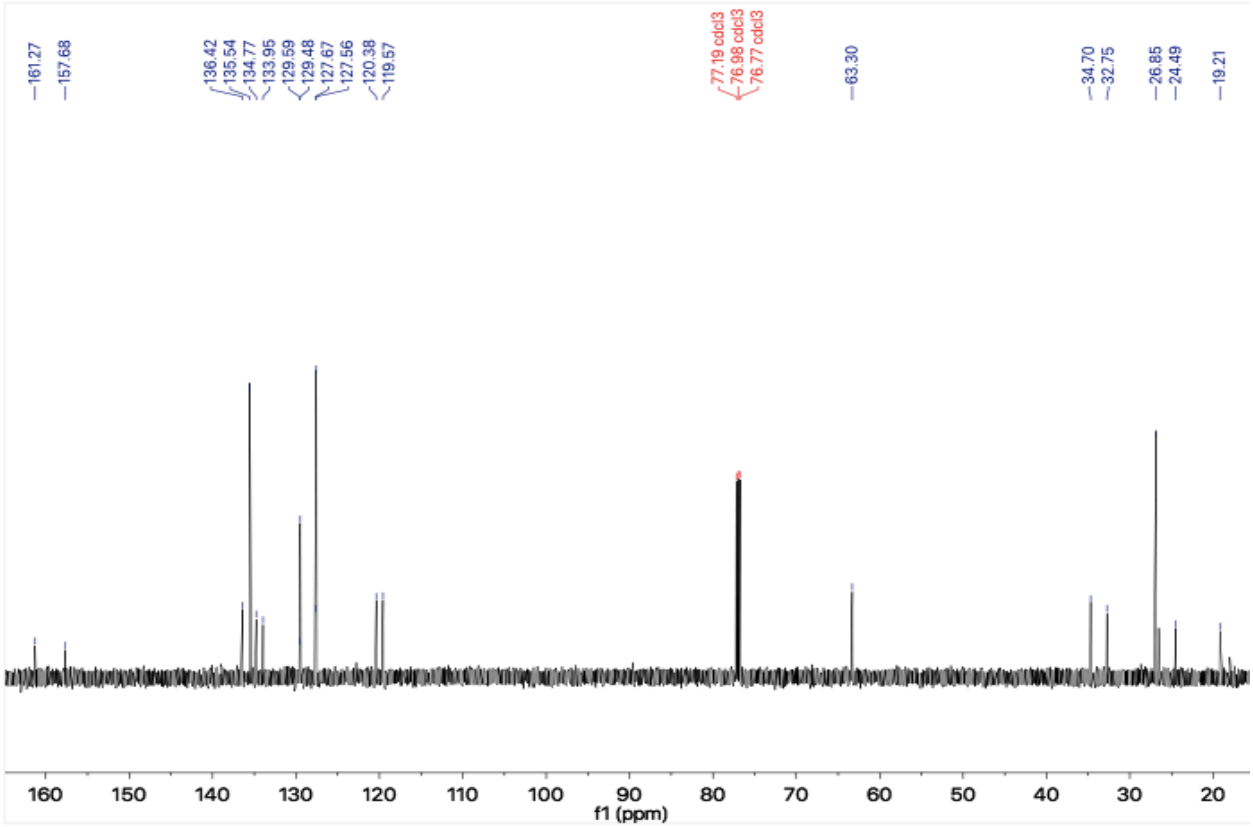
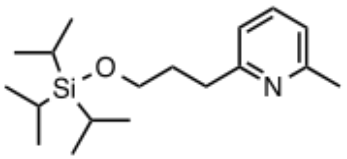
0.87 3.97 5.96 0.85 2.12 2.13 2.11 3.00 2.23 8.75
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


161.27 157.68 136.42 135.54 134.77 133.95 129.59 129.48 127.67 127.56 120.38 119.57 77.19 cdcl3 76.98 cdcl3 76.77 cdcl3 63.30 34.70 32.75 26.85 24.49 19.21
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)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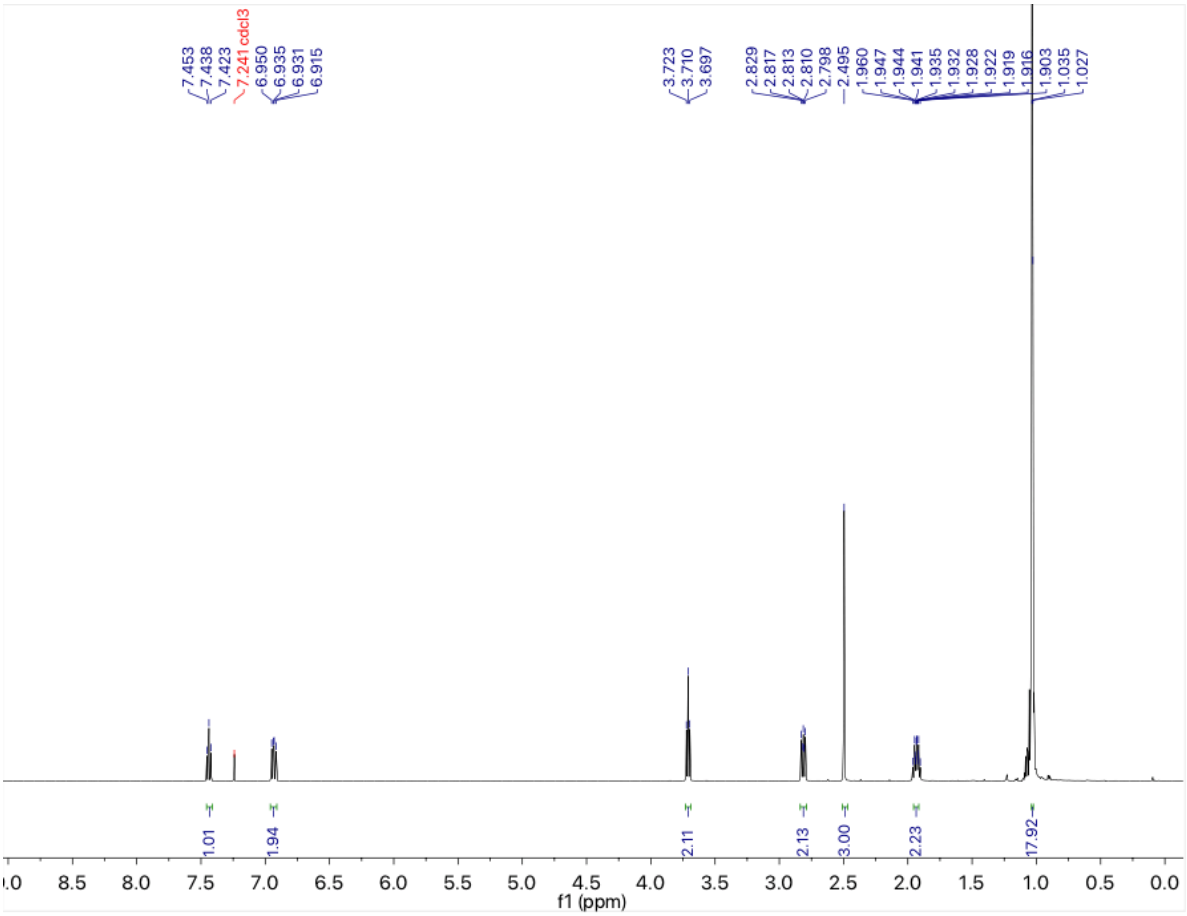
O
N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3.723
3.710
3.697
2.829
2.817
2.813
2.810
2.798
2.495
1.960
1.947
1.944
1.941
1.935
1.932
1.928
1.922
1.919
1.916
1.903
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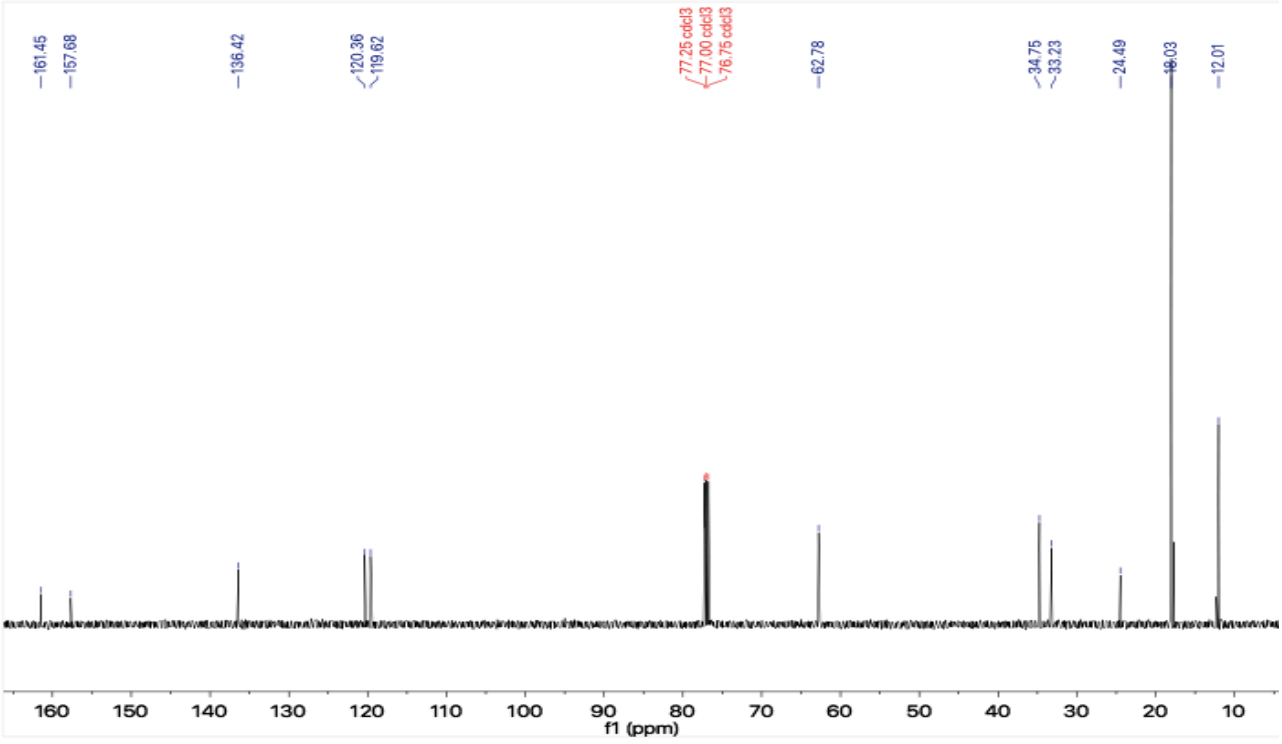
1.027
1.01
1.94
2.11
2.13
3.00
2.23
17.92
f1 (ppm)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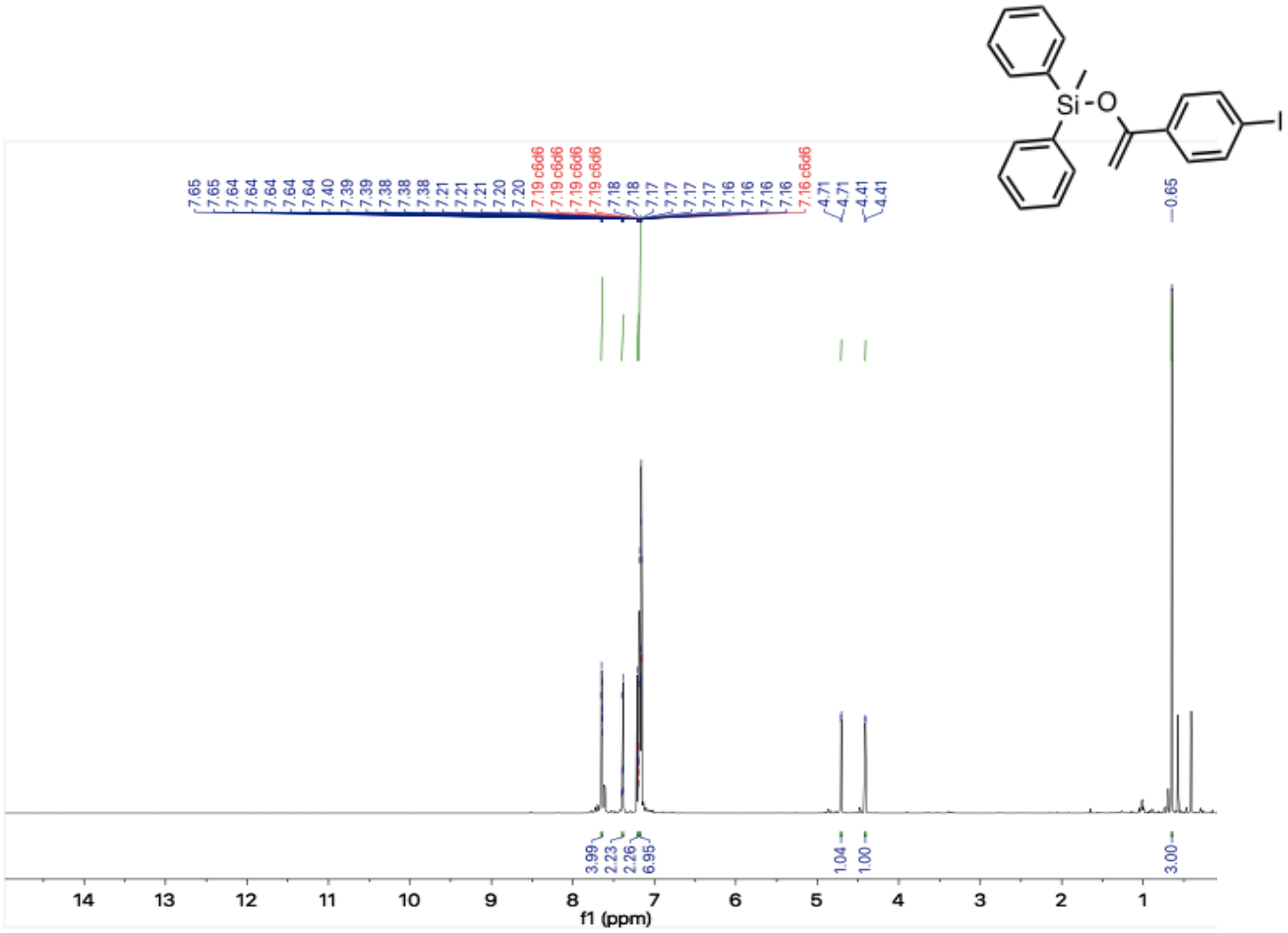
62.78
34.75
33.23
24.49
18.03
12.01
f1 (ppm)


Si
O
I
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7.39
7.38
7.21
7.20
7.19 c6d6
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7.17
7.16
4.71
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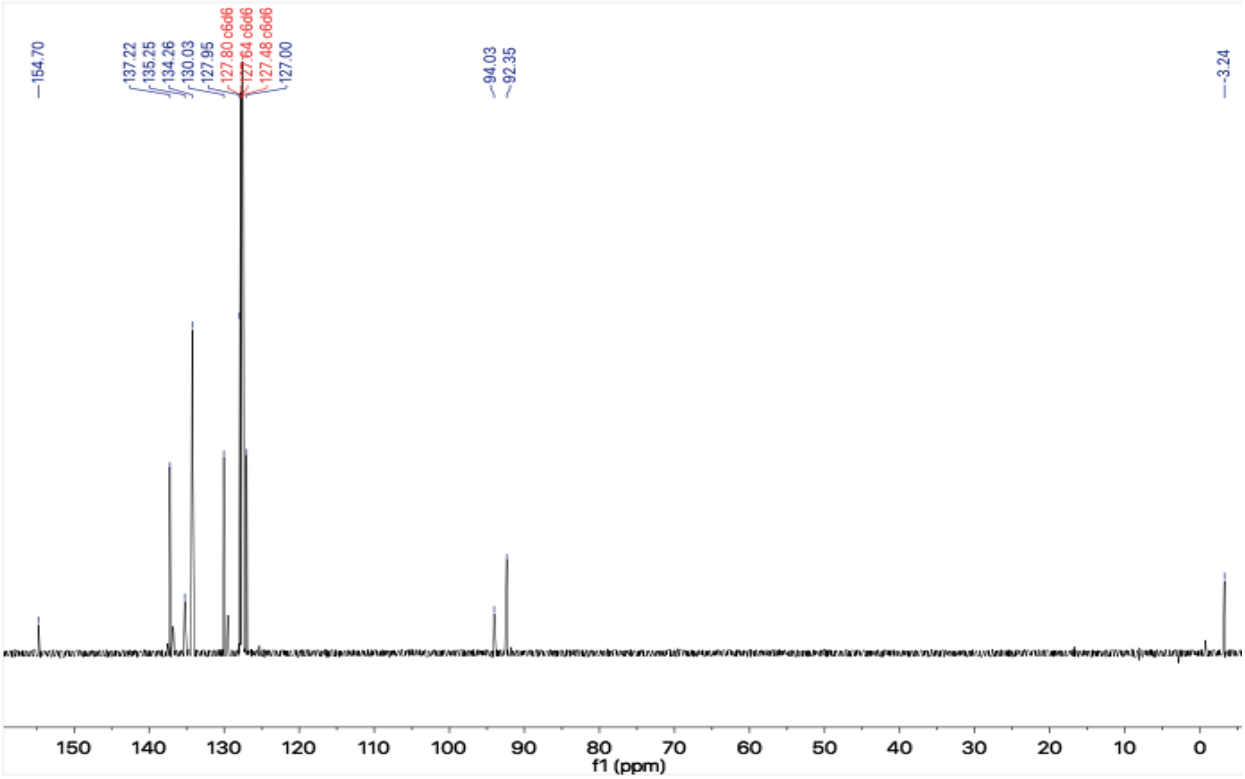
2.23
2.26
6.95
1.04
1.00
3.00
f1 (ppm)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135.25
134.26
130.03
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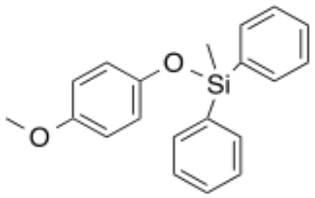
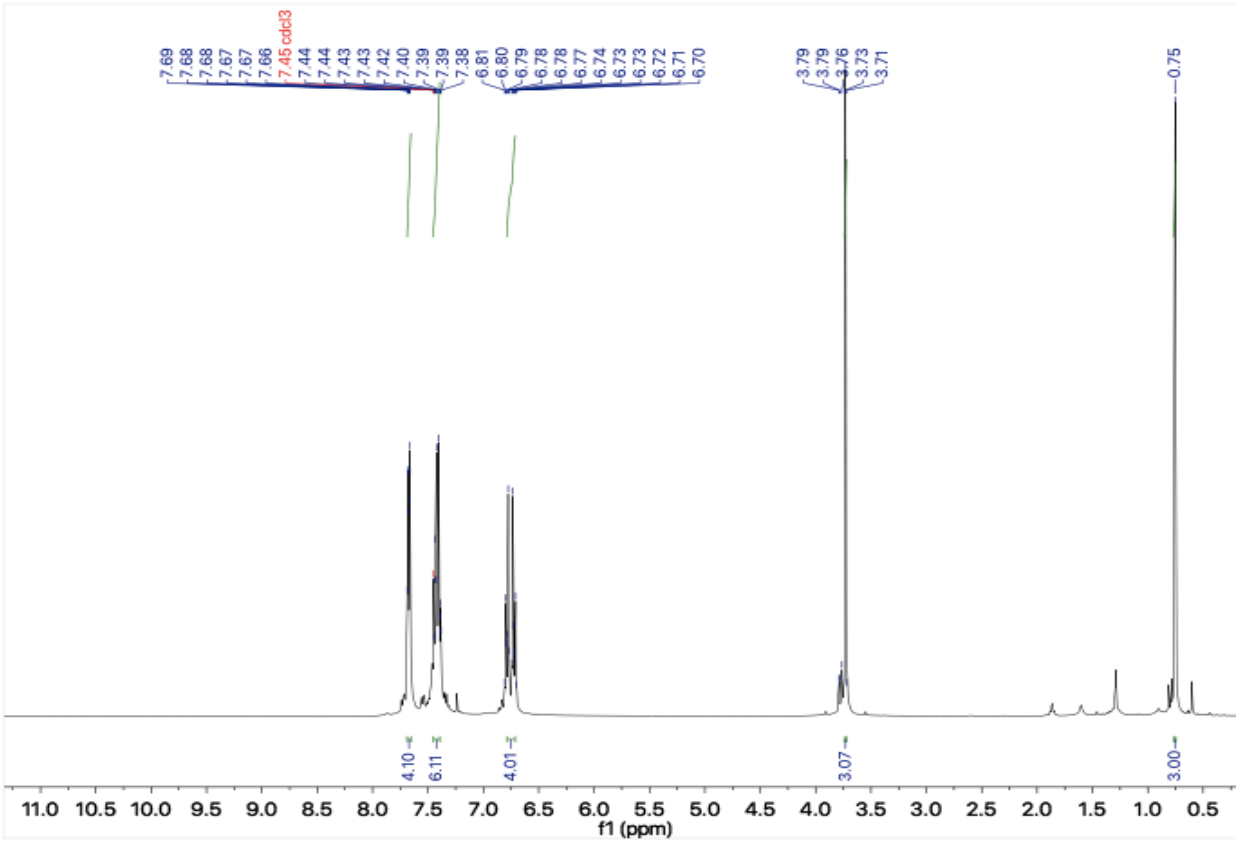
127.80 c6d6
127.64 c6d6
127.48 c6d6
127.00
94.03
92.35
3.24
f1 (ppm)


O
Si
O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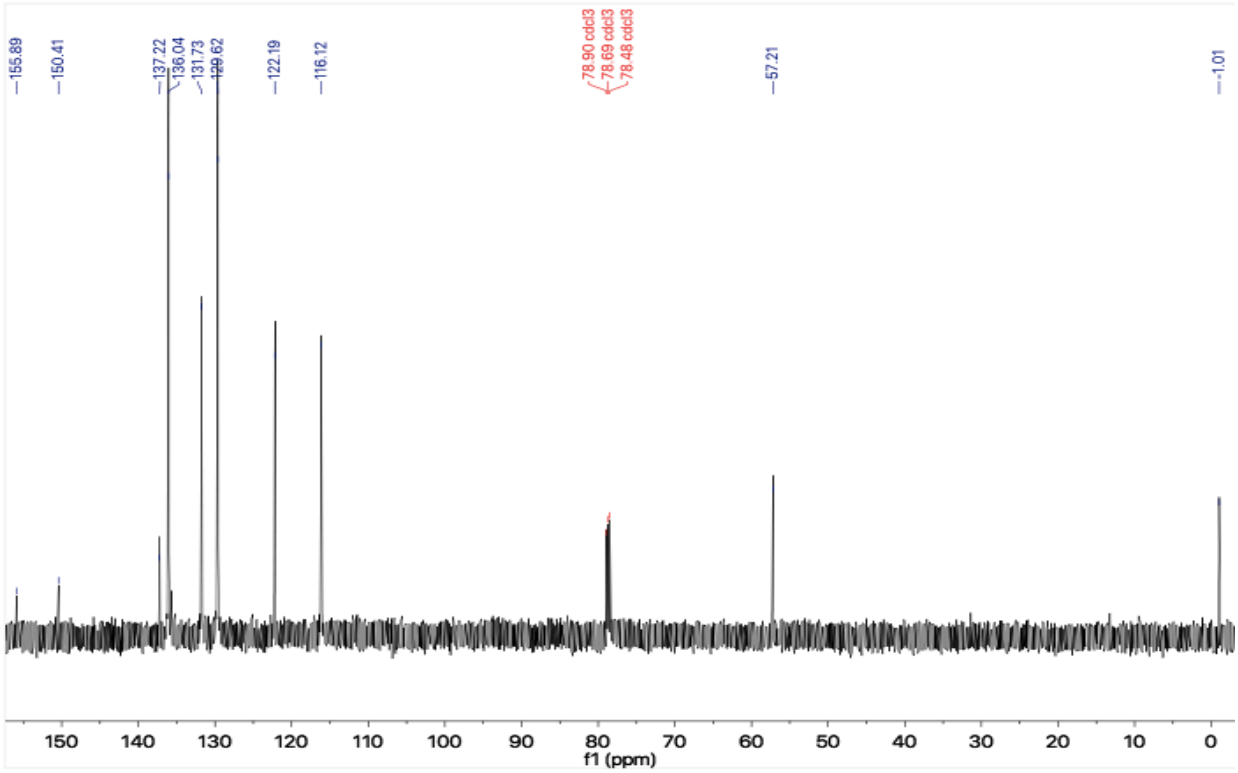
6.77
6.74
6.73
6.73
6.72
6.71
6.70
3.79
3.79
3.76
3.73
3.71
0.75
4.10
6.11
4.01
3.07
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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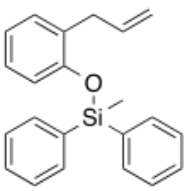
136.04
131.73
129.62
122.19
116.12
78.90 cdcl3
78.69 cdcl3
78.48 cdcl3
57.21
1.01
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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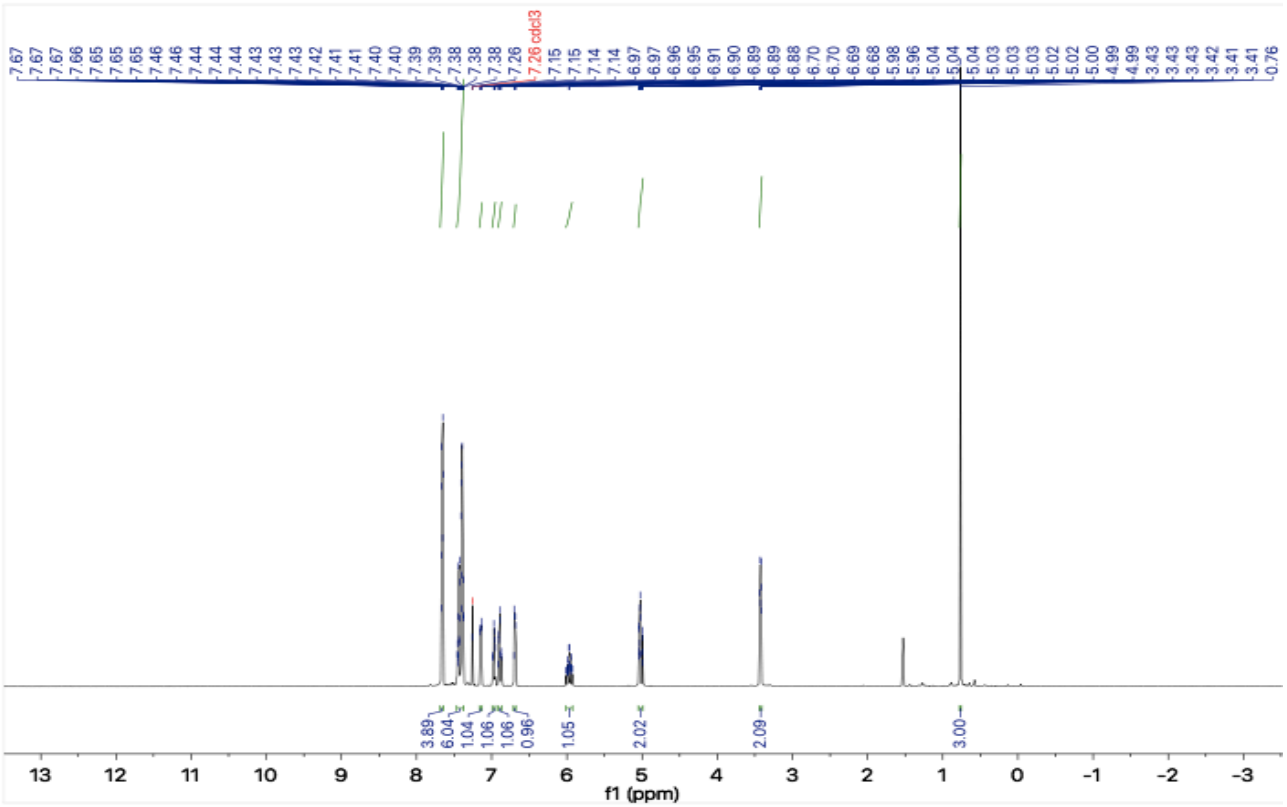
Si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3.89 6.04 1.04 1.06 1.06 0.96 1.05 2.02 2.09 3.00
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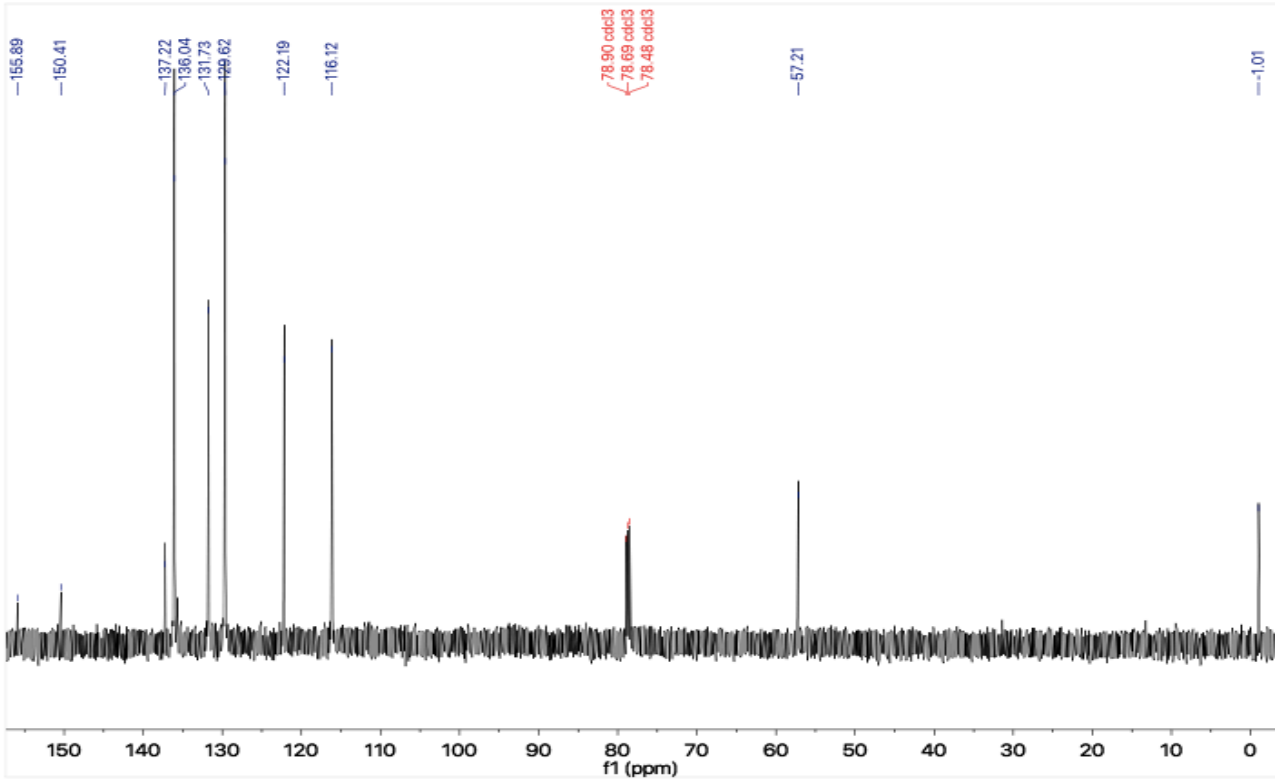
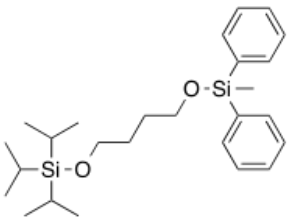
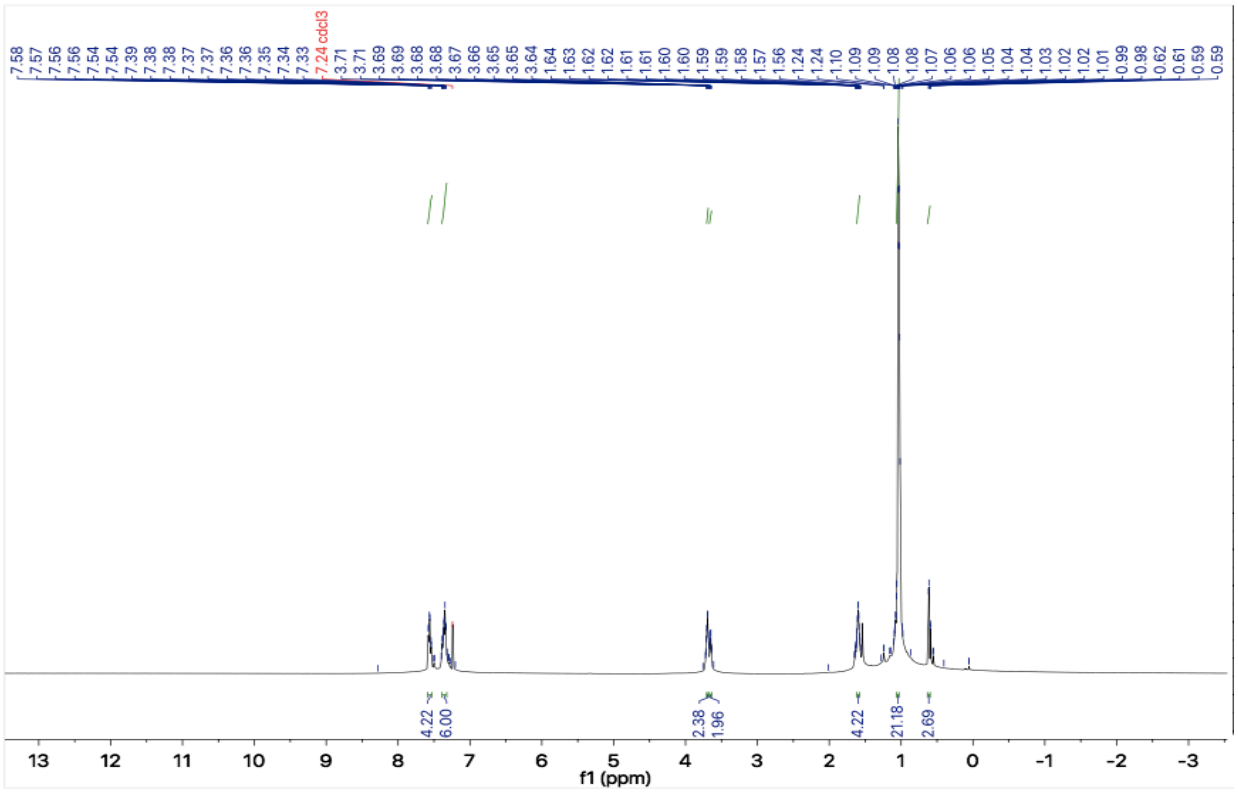
f1 (ppm)


155.89 150.41 137.22 136.04 131.73 129.62 122.19 116.12 78.90 cdcl3 78.69 cdcl3 78.48 cdcl3 57.21 -1.01
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O-Si
Si-O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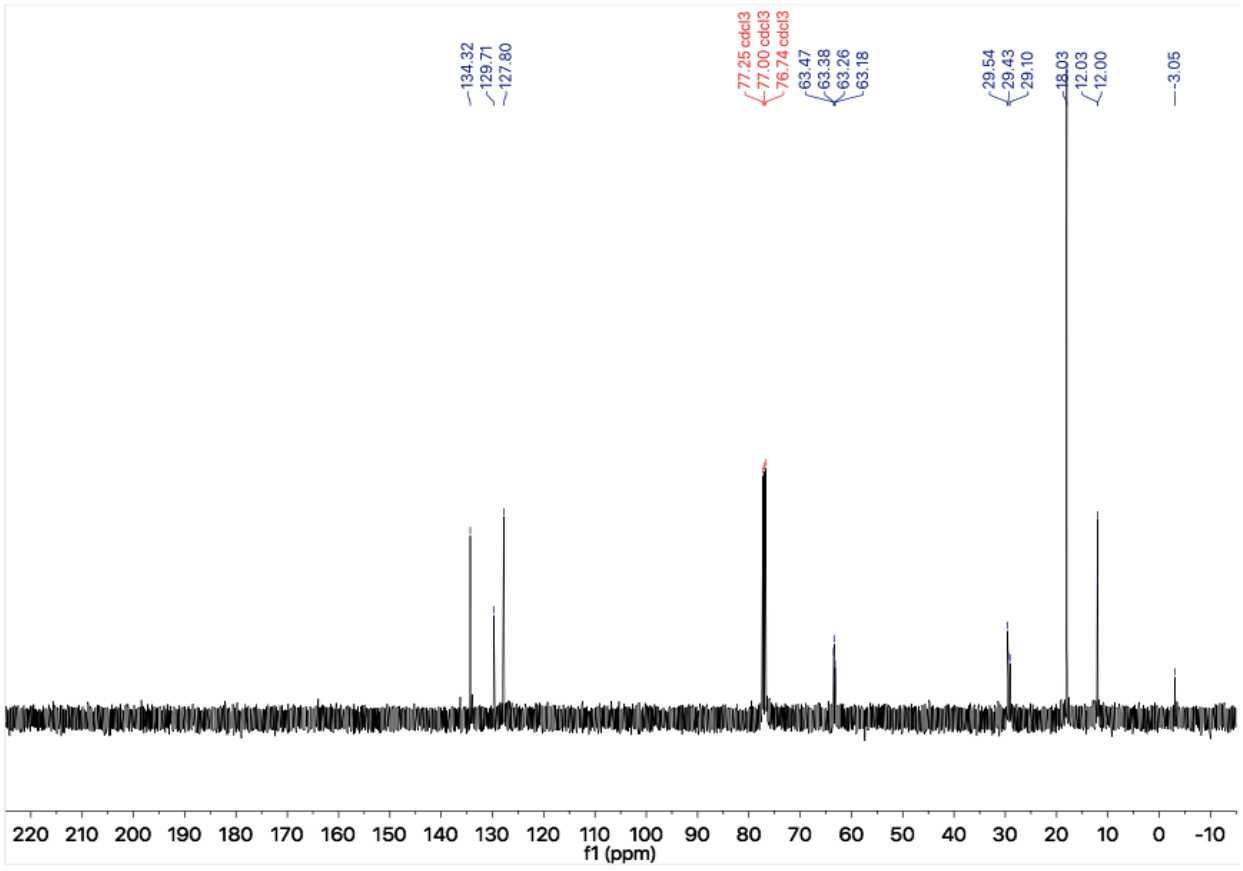
f1 (ppm)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77.25 cdcl3 77.00 cdcl3 76.74 cdcl3 63.47 63.38 63.26 63.18
29.54 29.43 29.10 18.03 12.03 12.00 -3.05
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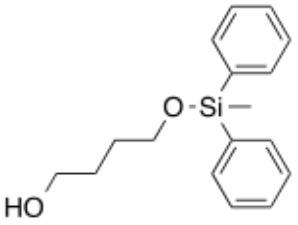
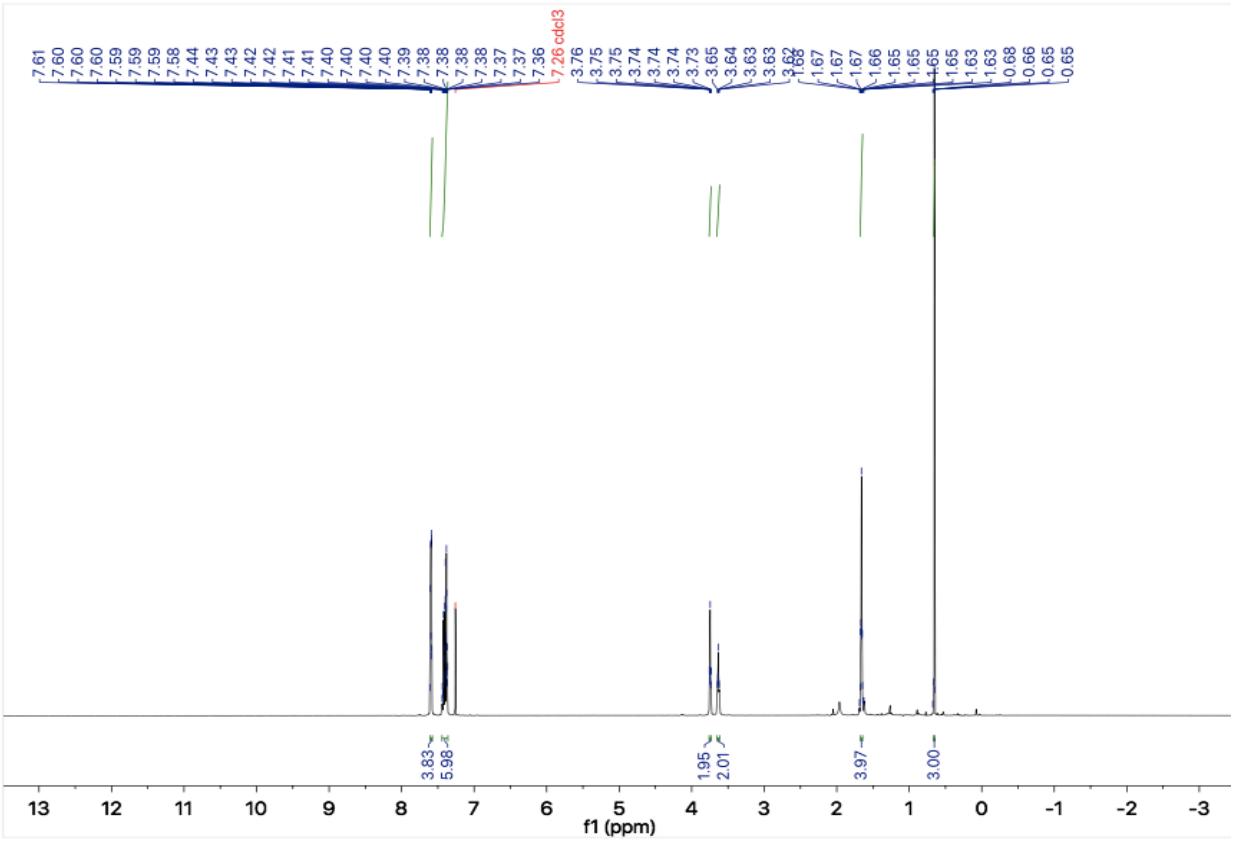
f1 (ppm)


O
Si
HO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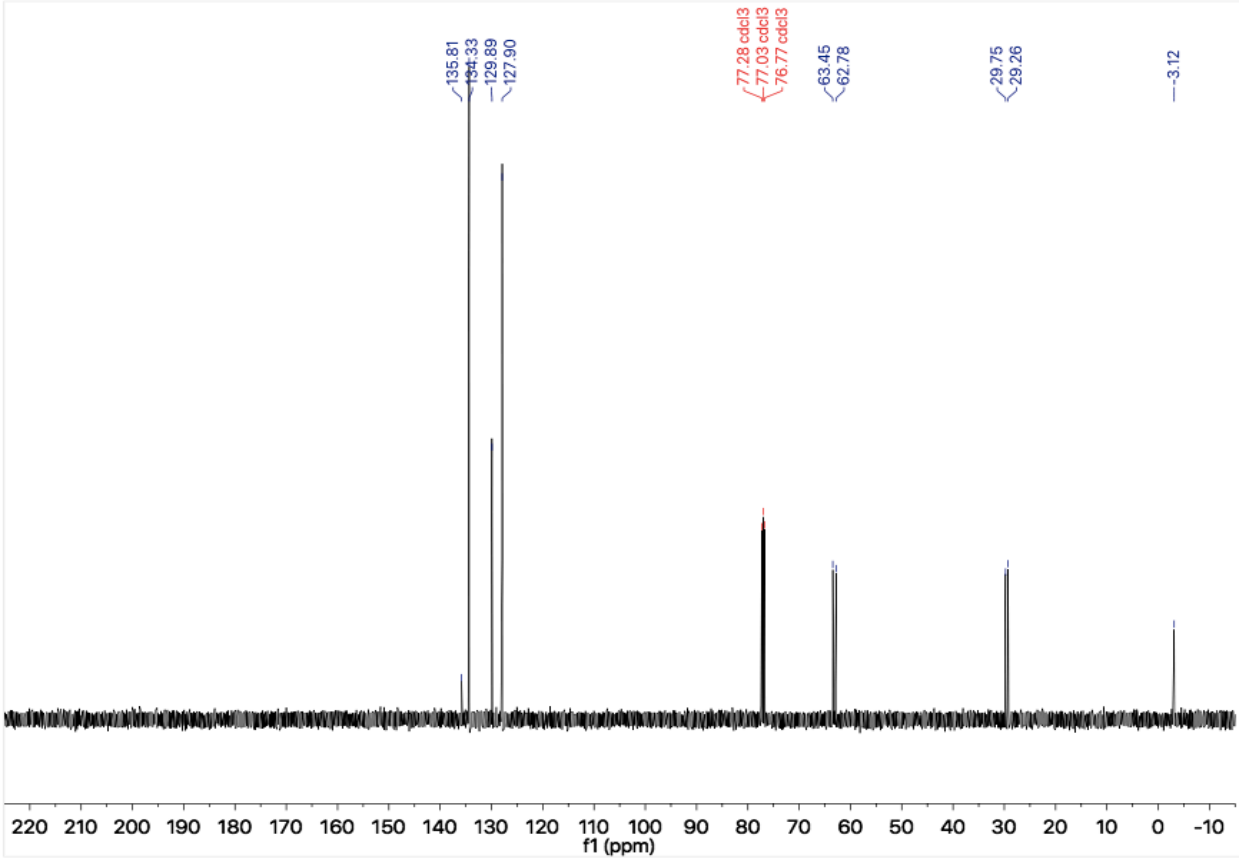
3.76 3.75 3.75 3.74 3.74 3.74 3.73 3.65 3.64 3.63 3.63 3.62 1.68 1.67 1.67 1.67 1.66 1.65 1.65 1.65 1.65 1.63 1.63 0.68 0.66 0.65 0.65
3.83 5.98 1.95 2.01 3.97 3.00
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)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29.75 29.26
-3.12
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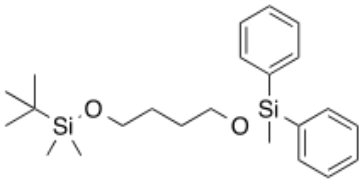
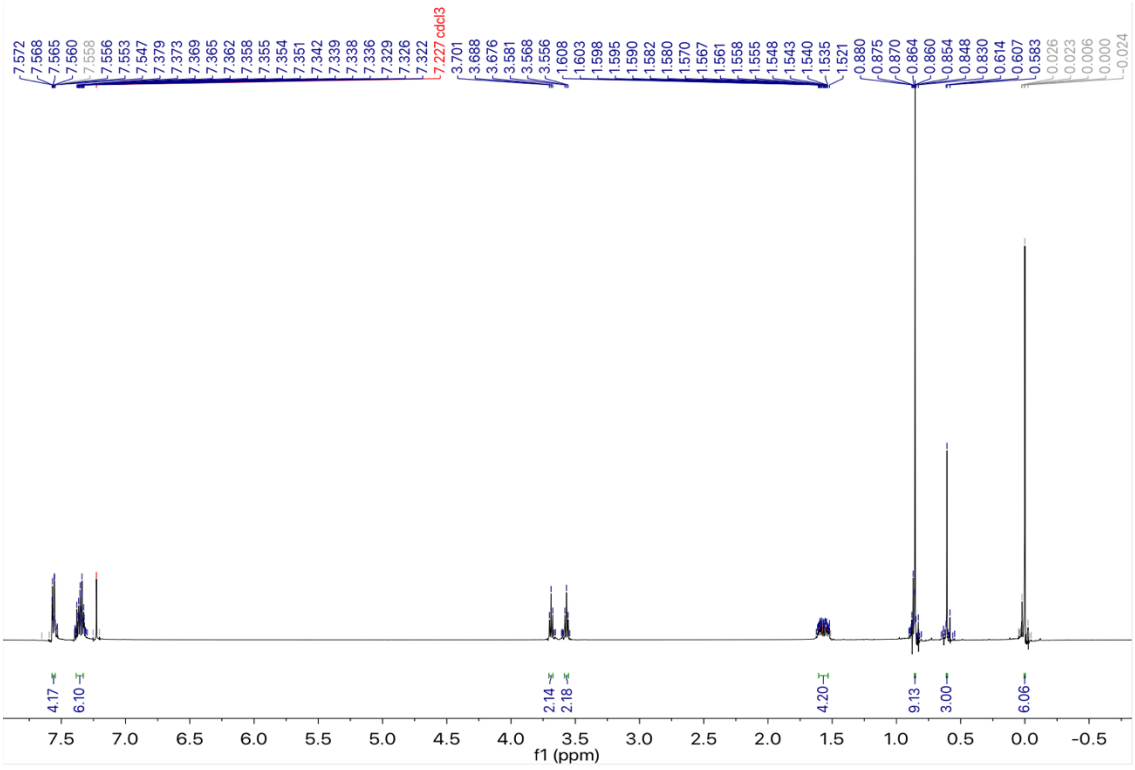
f1 (ppm)


Si
O
O
Si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7.322
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0.860
0.854
0.848
0.830
0.614
0.607
0.583
-0.026
-0.023
-0.006
0.000
-0.024
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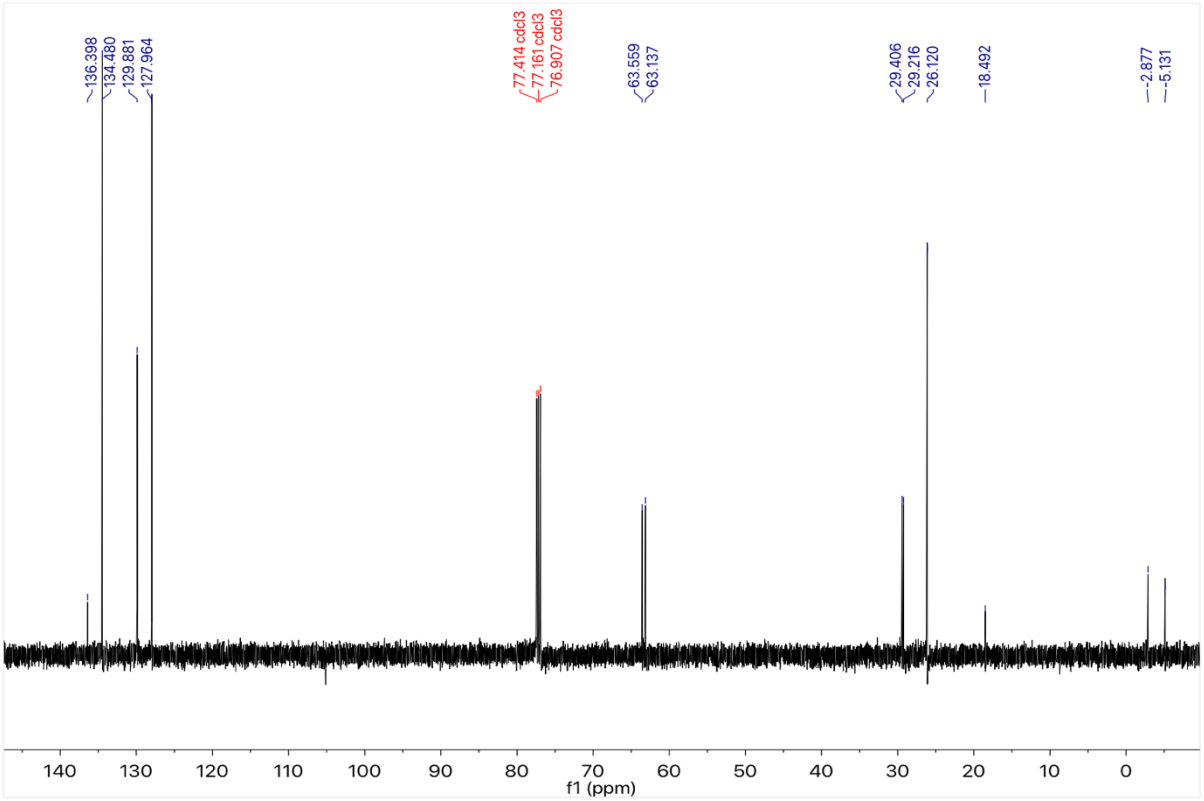
6.10
2.14
2.18
4.20
9.13
3.00
6.06
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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134.480
129.881
127.964
77.414 cdcl3
77.161 cdcl3
76.907 cdcl3
63.559
63.137
29.406
29.216
26.120
18.492
-2.877
-5.131
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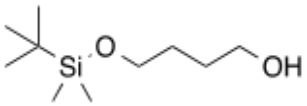
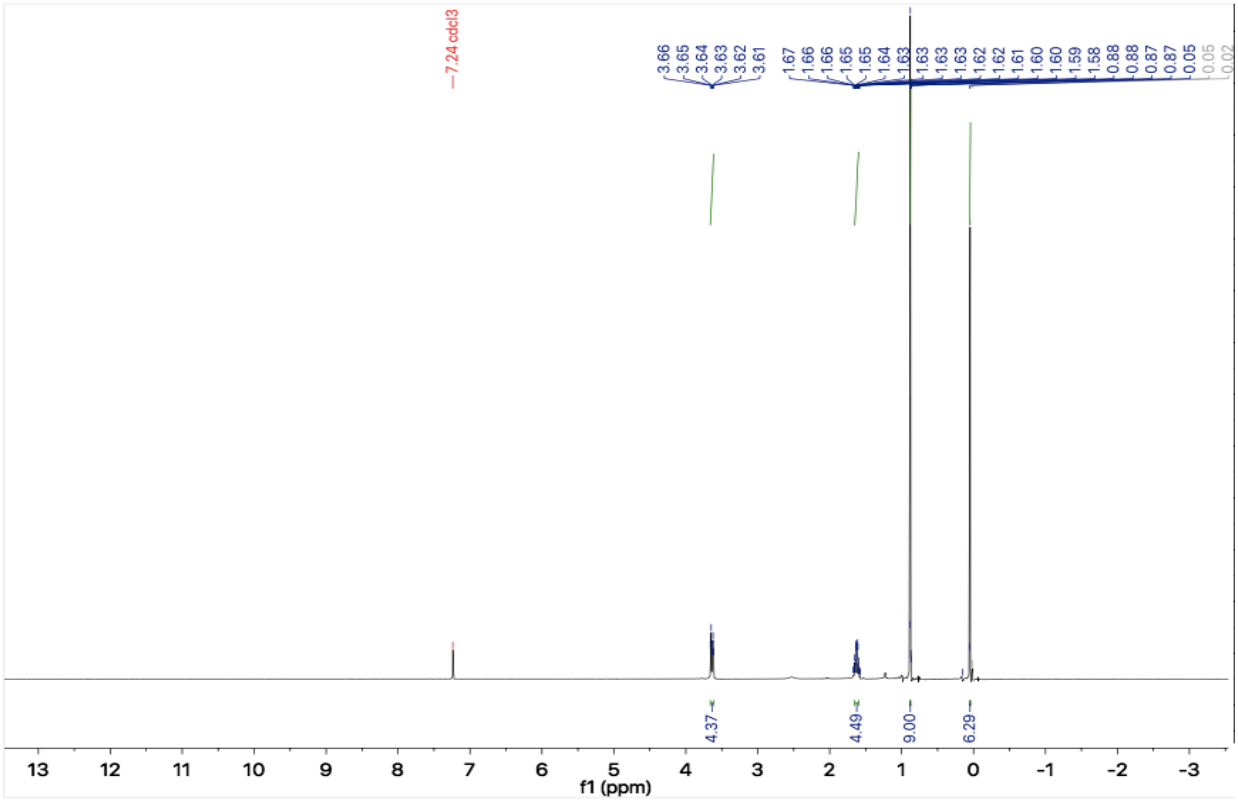
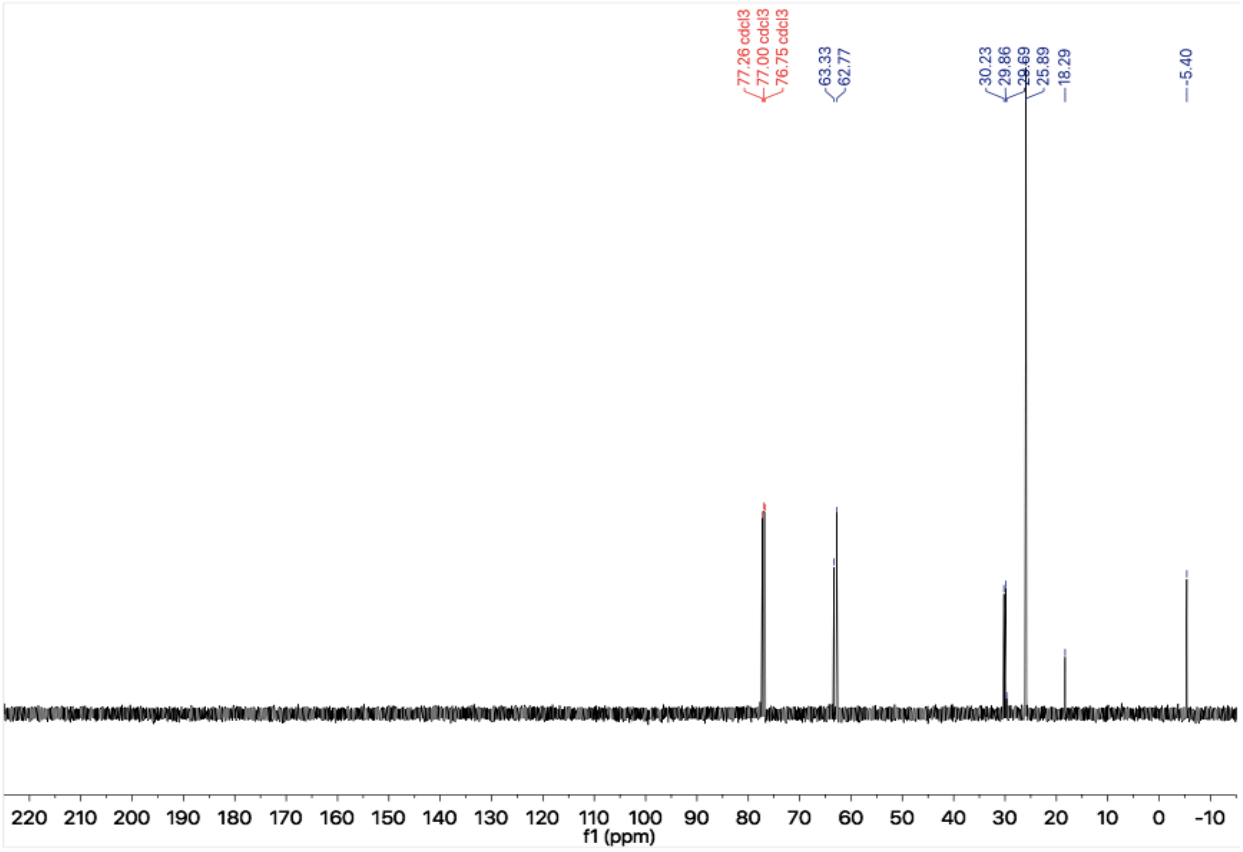
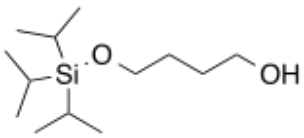
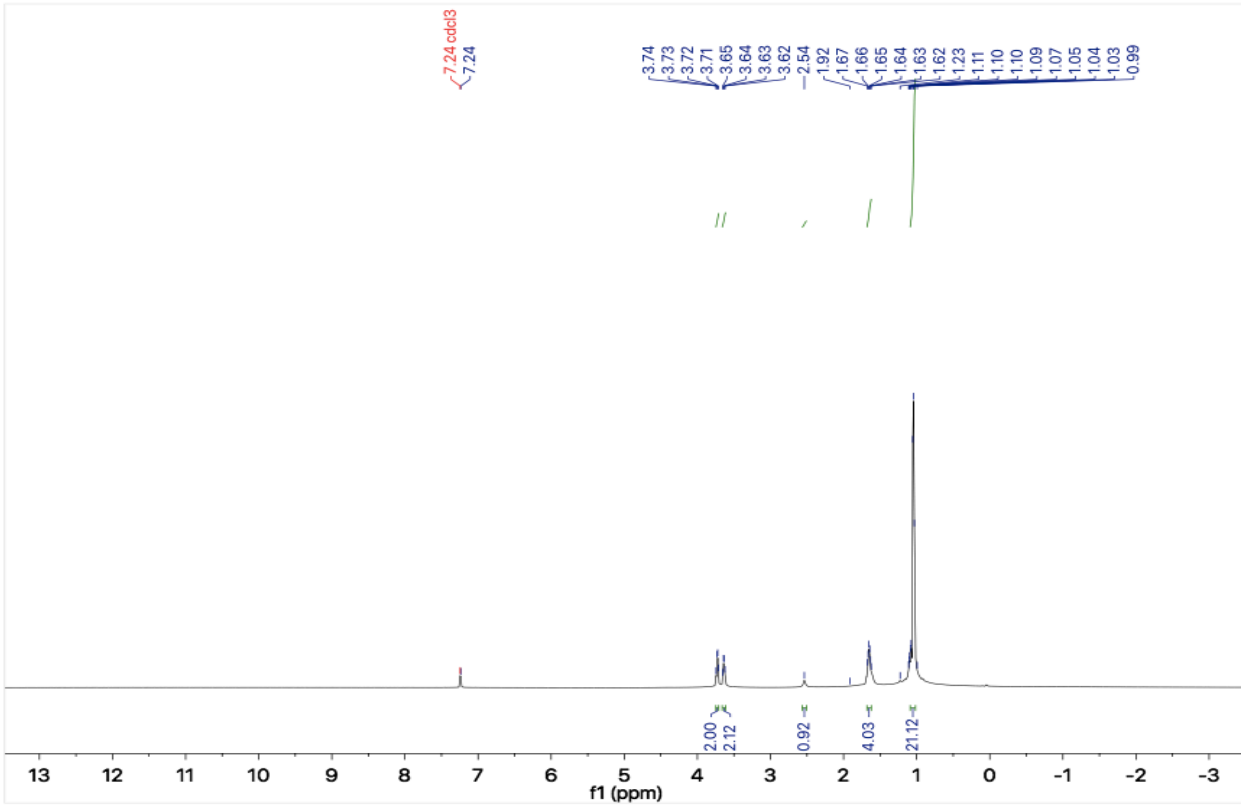
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

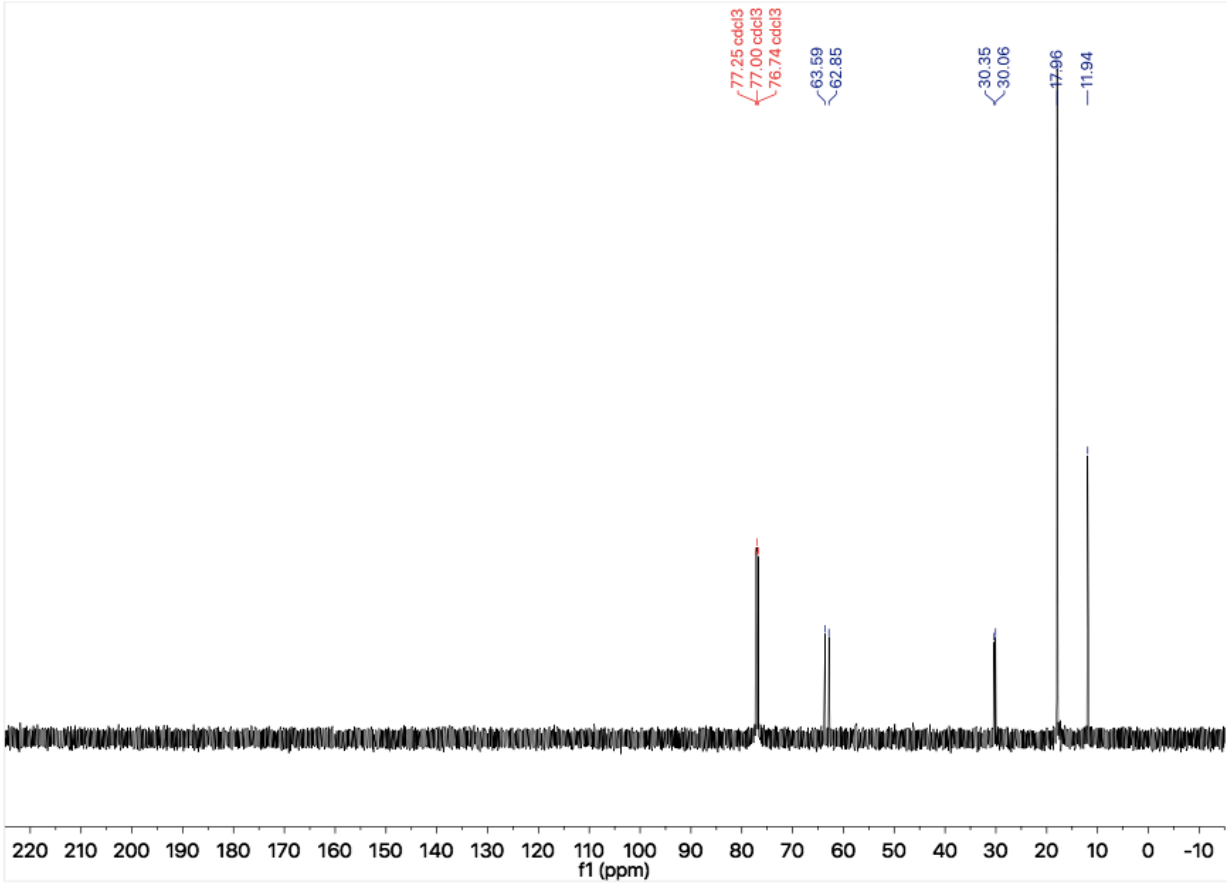

Si
O
OH


7.24 cdcl3
3.66
3.65
3.64
3.63
3.62
3.61
1.67
1.66
1.66
1.65
1.65
1.64
1.63
1.63
1.63
1.63
1.62
1.62
1.61
1.60
1.60
1.59
1.58
0.88
0.88
0.87
0.87
0.05
0.05
0.02
4.37
4.49
9.00
6.29
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)


77.26 cdcl3
77.00 cdcl3
76.75 cdcl3
63.33
62.77
30.23
29.86
29.69
25.89
18.29
-5.40
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Si
O
OH


7.24 cdcl3
7.24
3.74
3.73
3.72
3.71
3.65
3.64
3.63
3.62
2.54
1.92
1.67
1.66
1.65
1.64
1.63
1.62
1.23
1.11
1.10
1.10
1.09
1.07
1.05
1.04
1.03
0.99
2.00
2.12
0.92
4.03
21.12
f1 (ppm)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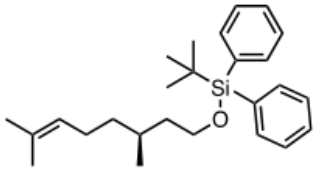
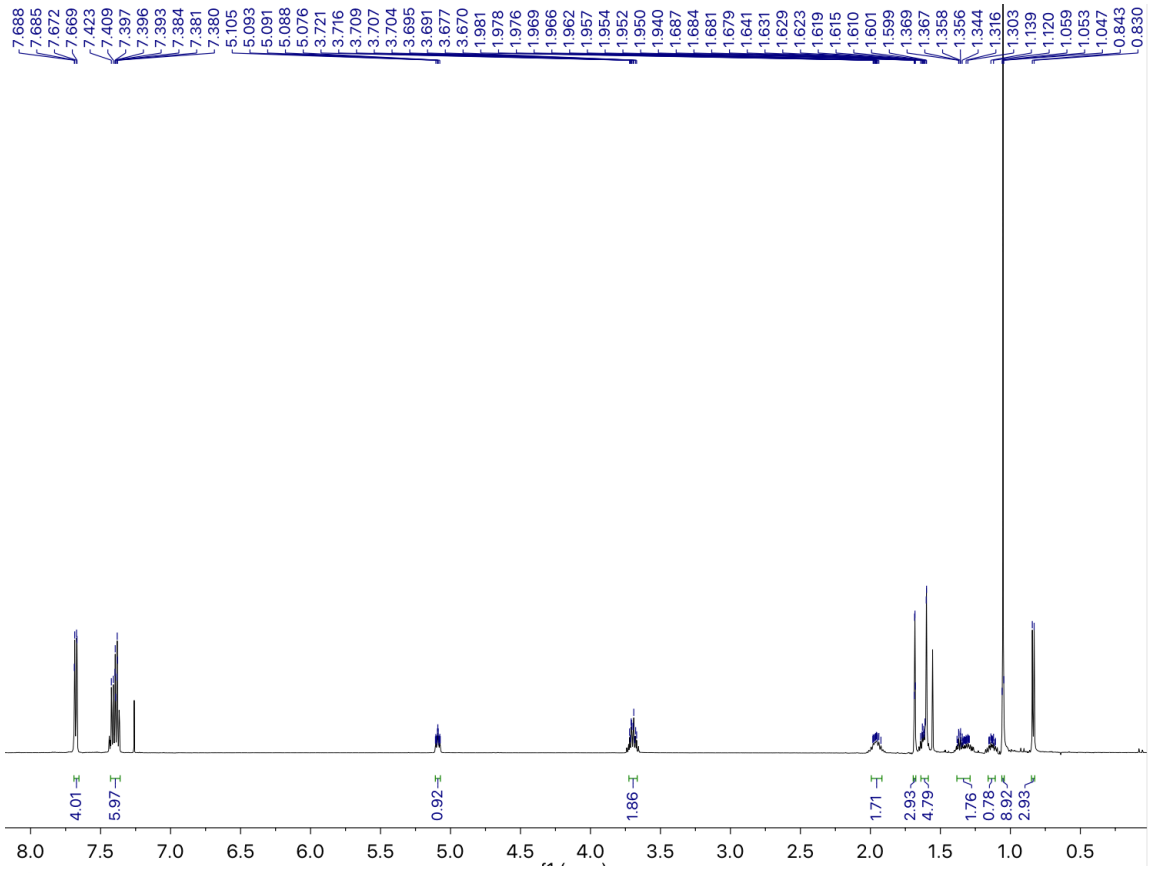
76.74 cdcl3
63.59
62.85
30.35
30.06
17.96
11.94
f1 (ppm)


Si
O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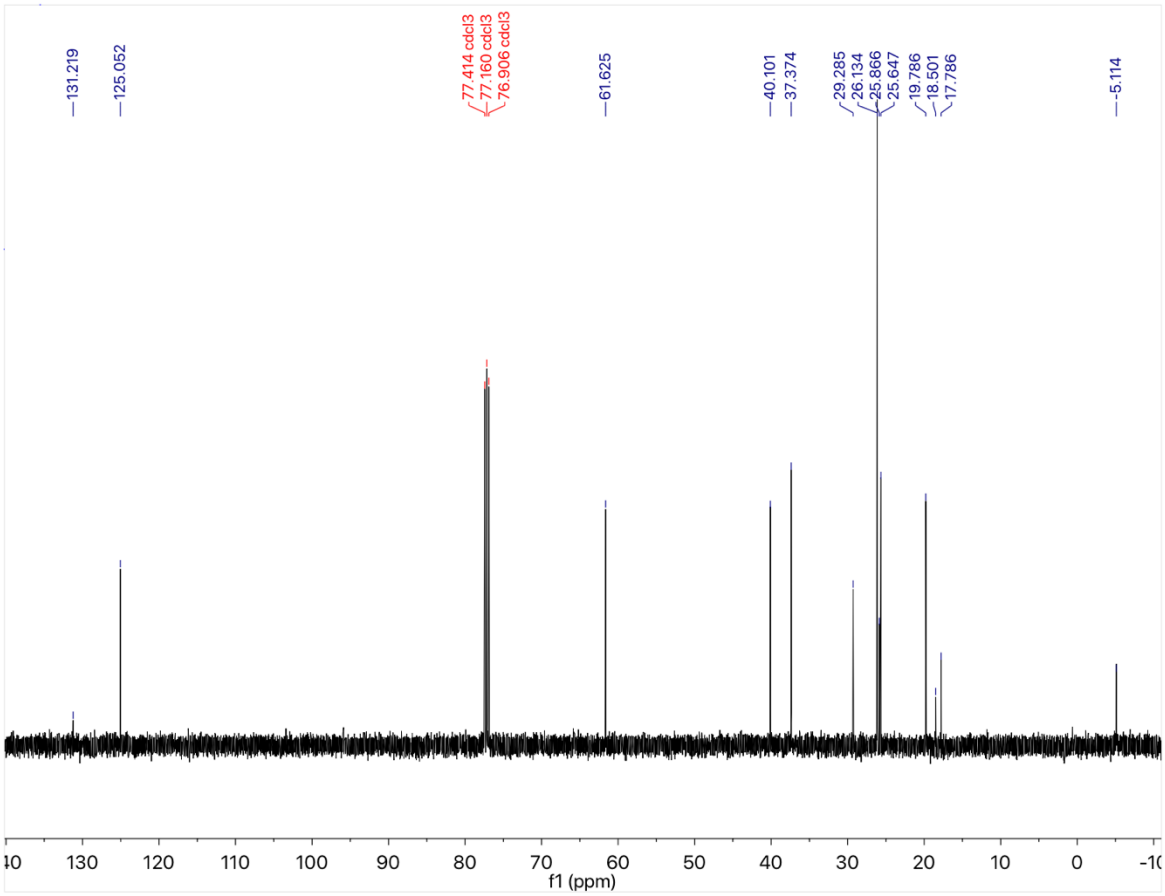
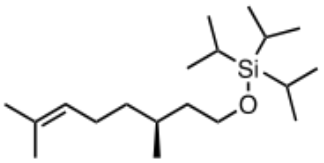
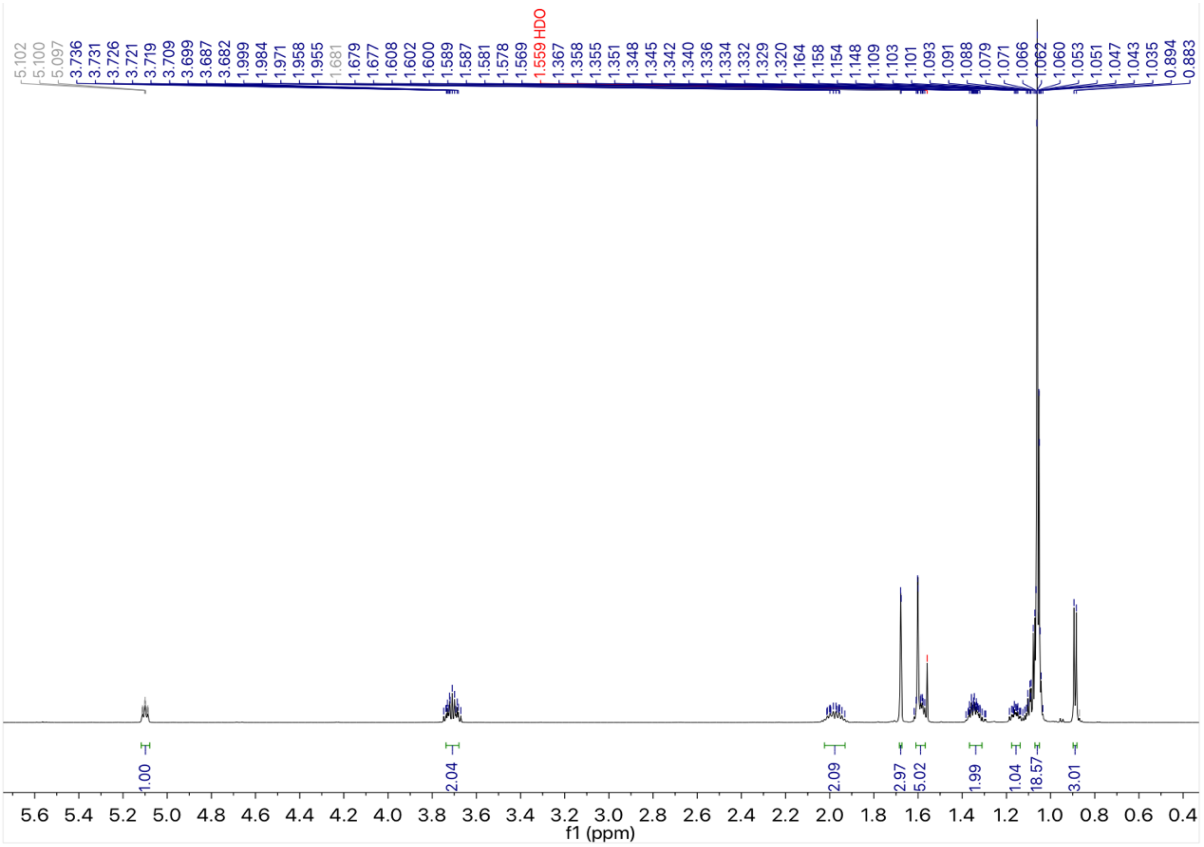
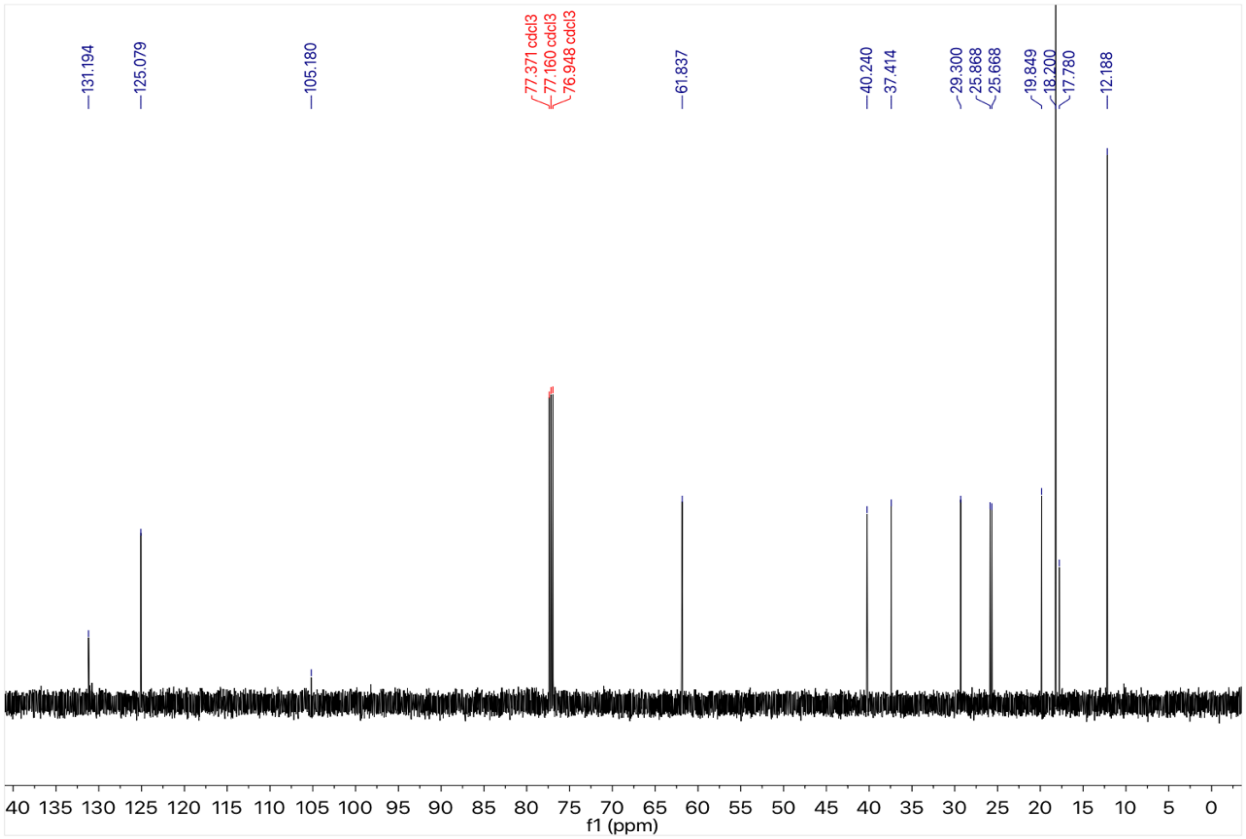
7.685
7.672
7.669
7.423
7.409
7.397
7.396
7.393
7.384
7.381
7.380
5.105
5.093
5.091
5.088
5.076
3.721
3.716
3.709
3.707
3.704
3.695
3.691
3.677
3.670
1.981
1.978
1.976
1.969
1.966
1.962
1.957
1.954
1.952
1.950
1.940
1.687
1.684
1.681
1.679
1.641
1.631
1.629
1.623
1.619
1.615
1.610
1.601
1.599
1.369
1.367
1.358
1.356
1.344
1.316
1.303
1.139
1.120
1.059
1.053
1.047
0.843
0.830
4.01
5.97
0.92
1.86
1.71
2.93
4.79
1.76
0.78
8.92
2.93
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5


131.219
125.052
77.414 cdcl3
77.160 cdcl3
76.906 cdcl3
61.625
40.101
37.374
29.285
26.134
25.866
25.647
19.786
18.501
17.786
-5.114
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)




5.102
5.100
5.097
3.736
3.731
3.726
3.721
3.719
3.709
3.699
3.687
3.682
1.999
1.984
1.971
1.958
1.955
1.681
1.679
1.677
1.608
1.602
1.600
1.589
1.587
1.581
1.578
1.569
1.559 HDO
1.367
1.358
1.355
1.351
1.348
1.345
1.342
1.340
1.336
1.334
1.332
1.329
1.320
1.164
1.158
1.154
1.148
1.109
1.103
1.101
1.093
1.091
1.088
1.079
1.071
1.066
1.062
1.060
1.053
1.051
1.047
1.043
1.035
0.894
0.883
1.00
2.04
2.09
2.97
5.02
1.99
1.04
18.57
3.01
5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2 1.0 0.8 0.6 0.4
f1 (ppm)


131.194
125.079
105.180
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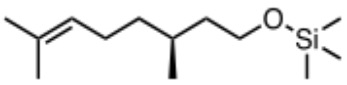
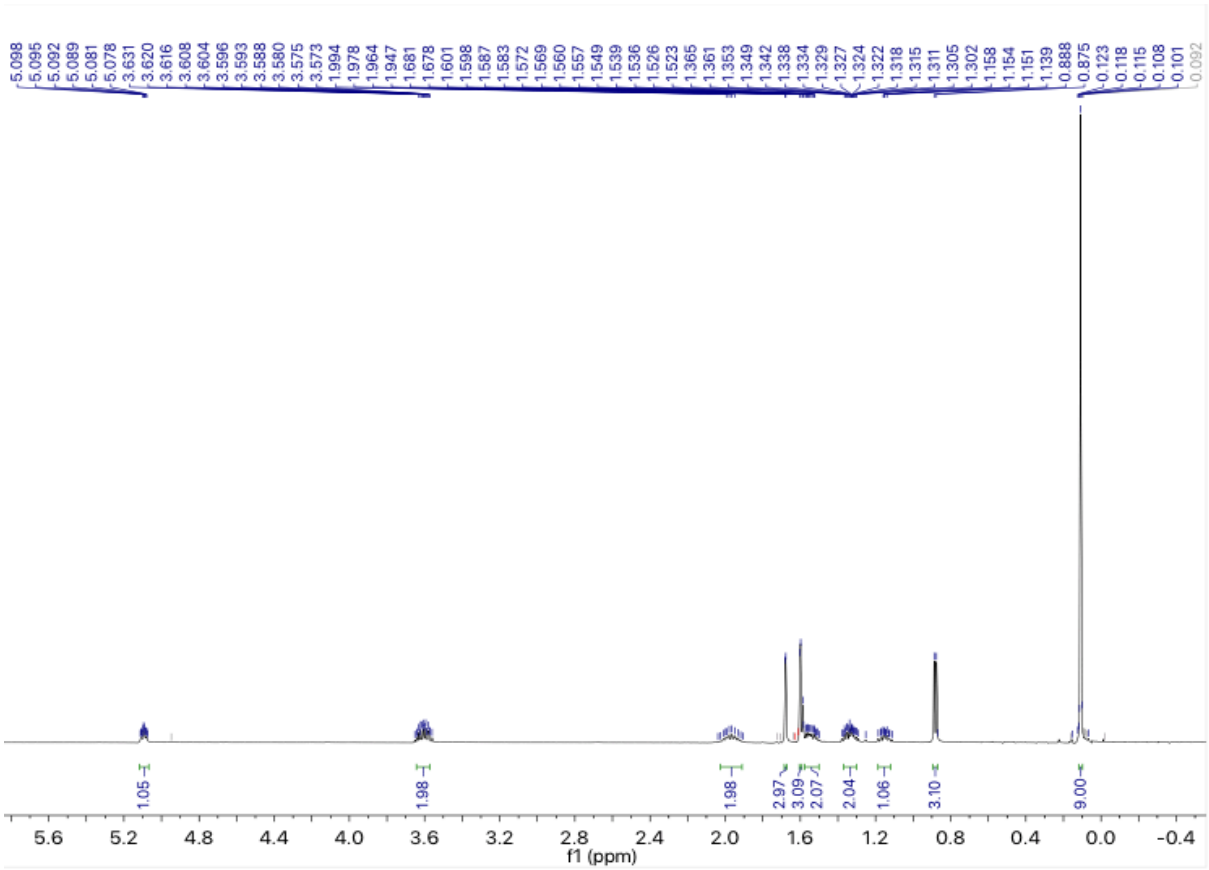
77.160 cdcl3
76.948 cdcl3
61.837
40.240
37.414
29.300
25.868
25.668
19.849
18.200
17.780
12.188
40 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)


O
Si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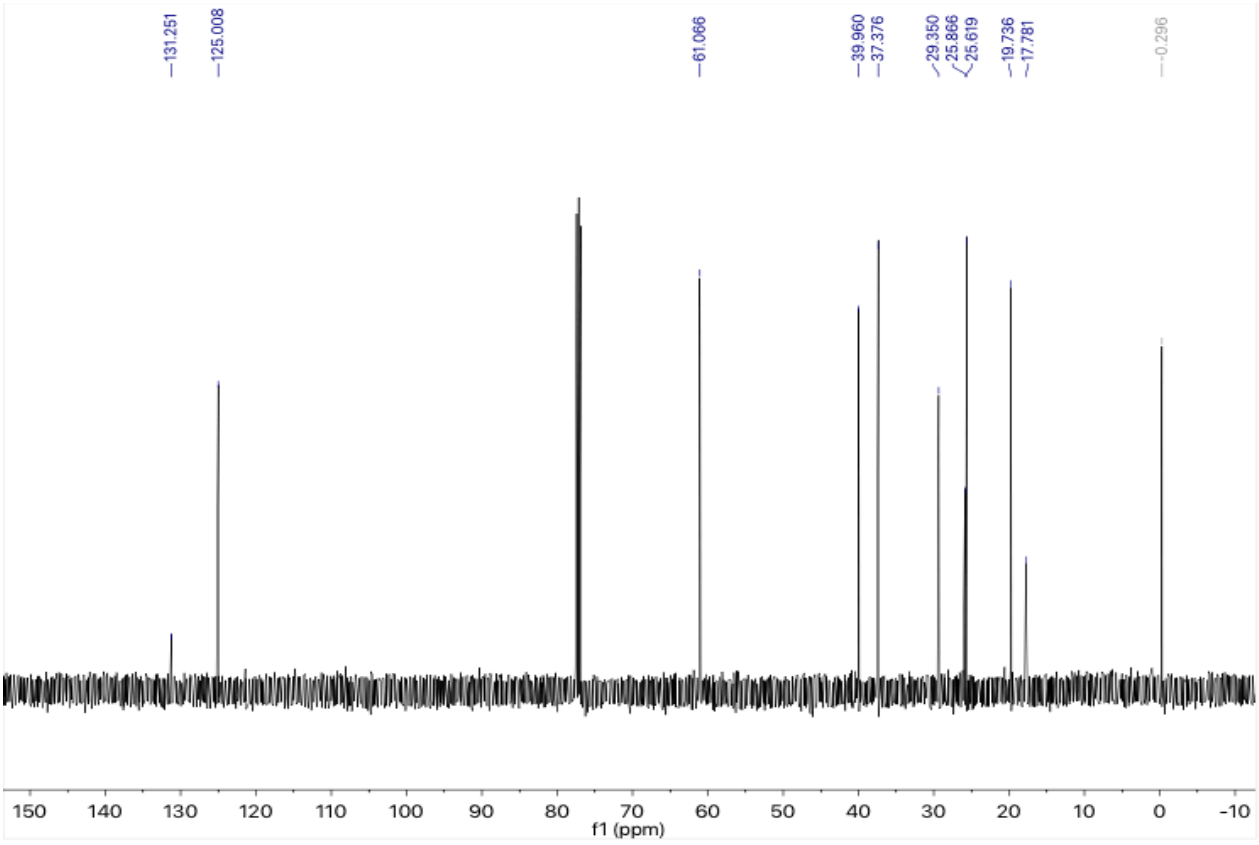
5.095
5.092
5.089
5.081
5.078
3.631
3.620
3.616
3.608
3.604
3.596
3.593
3.588
3.580
3.575
3.573
1.994
1.978
1.964
1.947
1.681
1.678
1.601
1.598
1.587
1.583
1.572
1.569
1.560
1.557
1.549
1.539
1.536
1.526
1.523
1.365
1.361
1.353
1.349
1.342
1.338
1.334
1.329
1.327
1.324
1.322
1.318
1.315
1.311
1.305
1.302
1.158
1.154
1.151
1.139
0.888
0.875
0.123
0.118
0.115
0.108
0.101
0.092
1.05
1.98
1.98
2.97
3.09
2.07
2.04
1.06
3.10
9.00
5.6
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
0.8
0.4
0.0
-0.4
f1 (ppm)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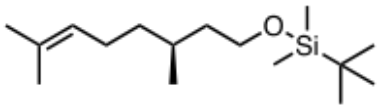
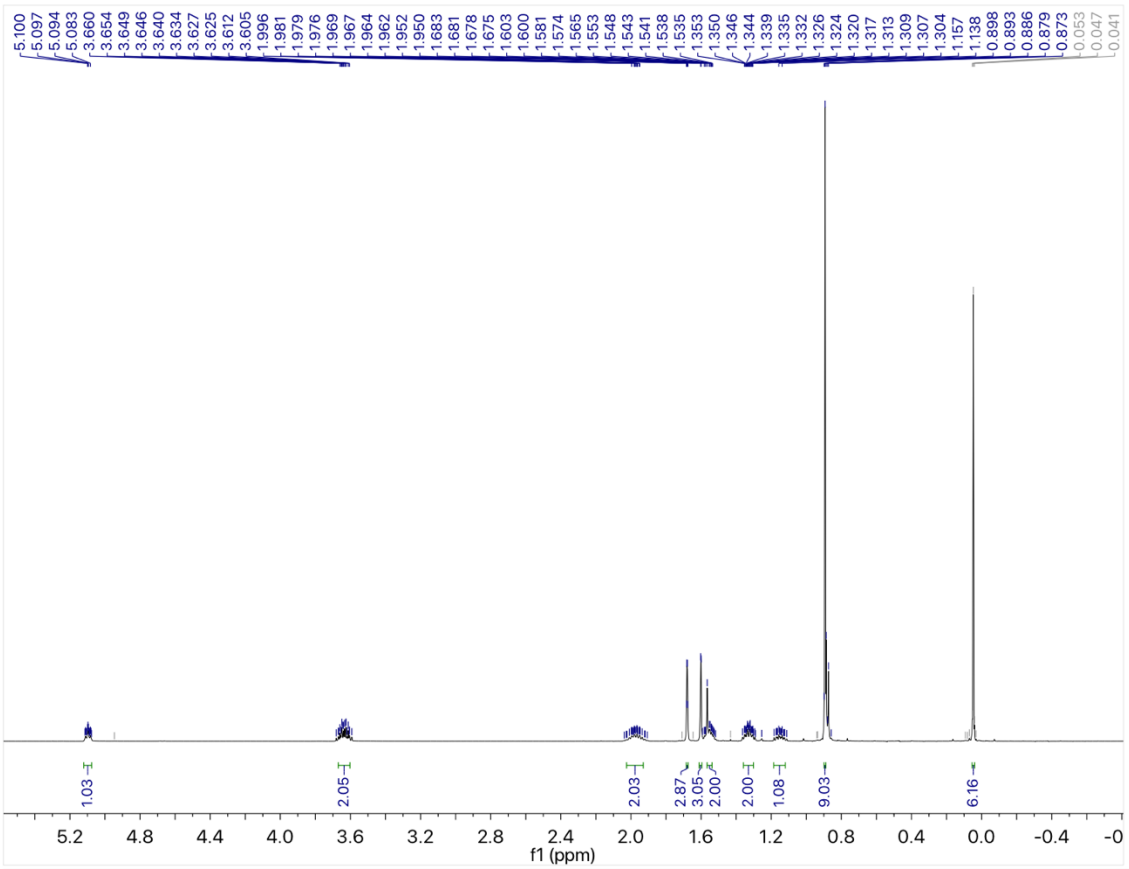
125.008
61.066
39.960
37.376
29.350
25.866
25.619
19.736
17.781
0.296
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

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5.097
5.094
5.083
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3.654
3.649
3.646
3.640
3.634
3.627
3.625
3.612
3.605
1.996
1.981
1.979
1.976
1.969
1.967
1.964
1.962
1.952
1.950
1.683
1.681
1.678
1.675
1.603
1.600
1.581
1.574
1.565
1.553
1.548
1.543
1.541
1.538
1.535
1.353
1.350
1.346
1.344
1.339
1.335
1.332
1.326
1.324
1.320
1.317
1.313
1.309
1.307
1.304
1.157
1.138
0.898
0.893
0.886
0.879
0.873
0.053
0.047
0.041
1.03
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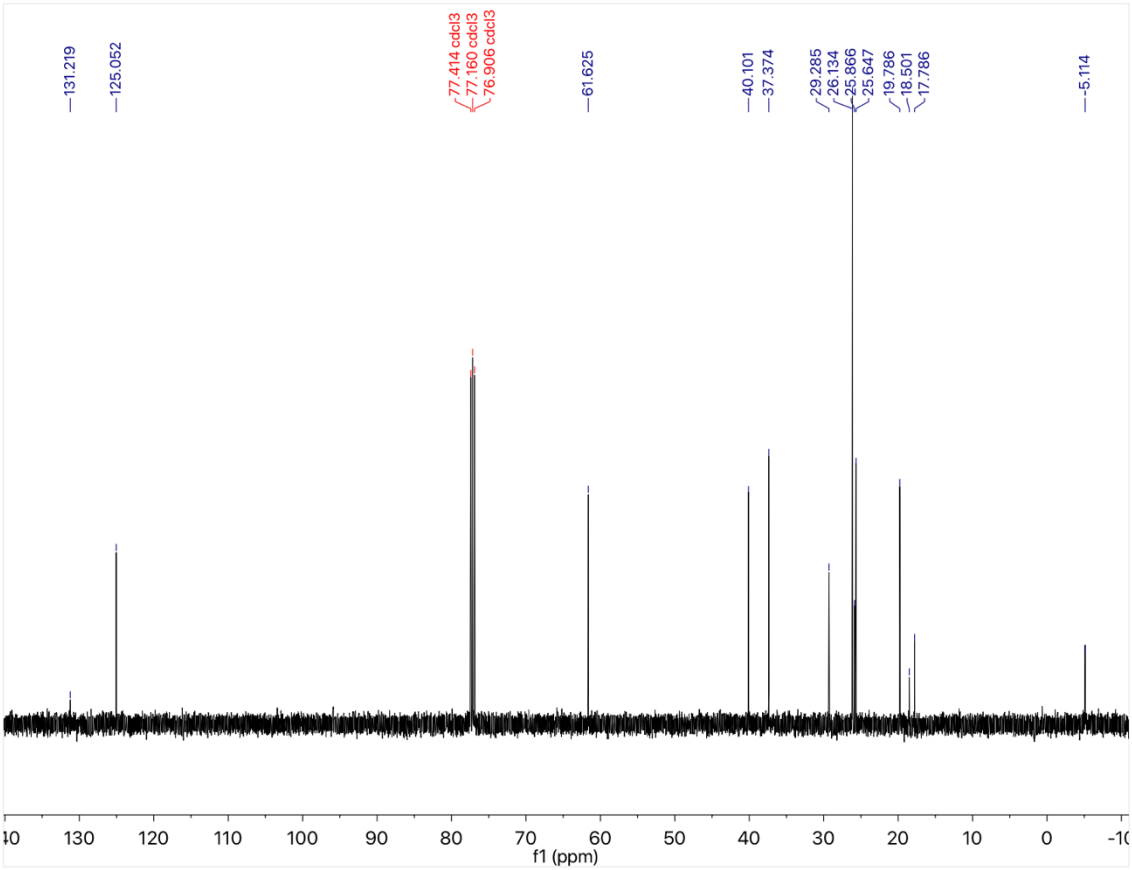
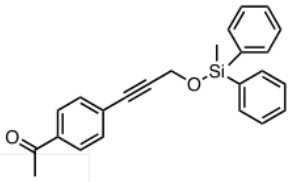
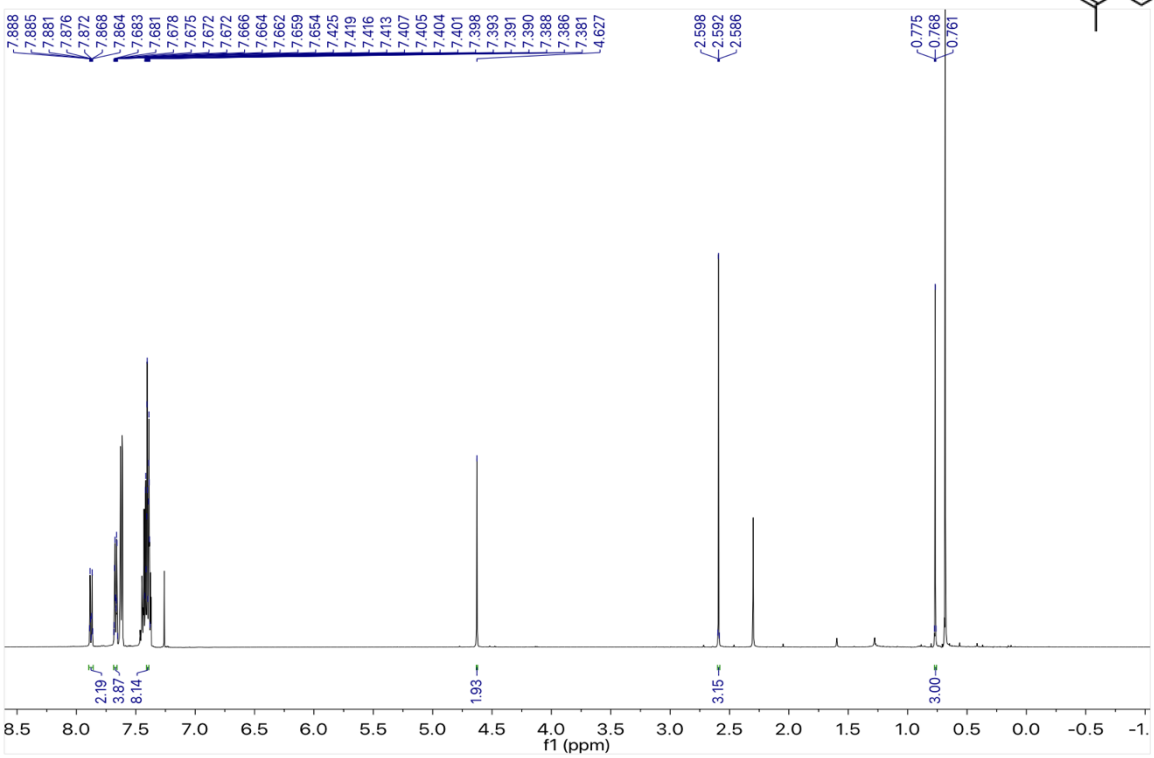
2.03
2.87
3.05
2.00
2.00
1.08
9.03
6.16
f1 (ppm)


131.219
125.052
77.414 cdcl3
77.160 cdcl3
76.906 cdcl3
61.625
40.101
37.374
29.285
26.134
25.866
25.647
19.786
18.501
17.786
-5.114
f1 (ppm)




7.888
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7.881
7.876
7.872
7.868
7.864
7.683
7.681
7.678
7.675
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7.672
7.666
7.664
7.662
7.659
7.654
7.425
7.419
7.416
7.413
7.407
7.405
7.404
7.401
7.398
7.393
7.391
7.390
7.388
7.386
7.381
4.627
2.598
2.592
2.586
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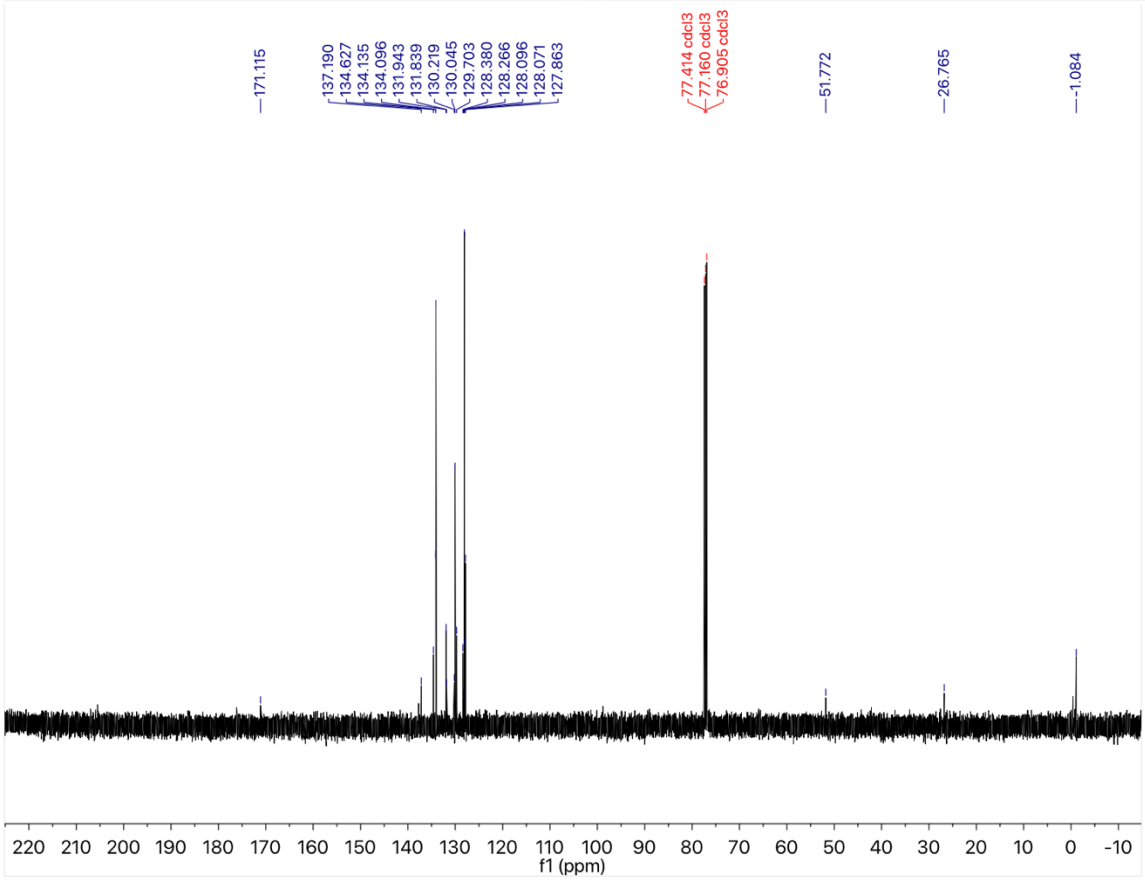
0.768
0.761
2.19
3.87
8.14
1.93
3.15
3.00
f1 (ppm)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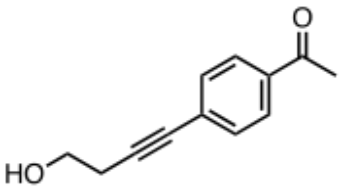
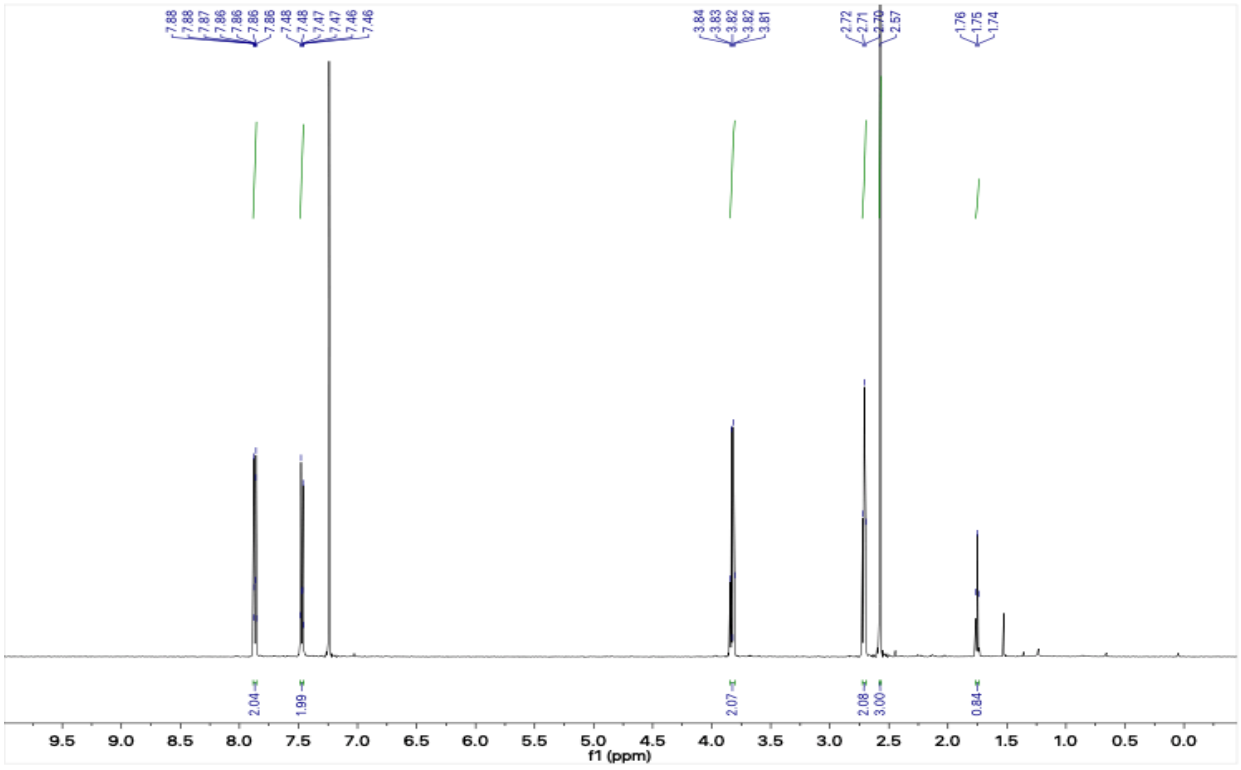
134.135
134.096
131.943
131.839
130.219
130.045
129.703
128.380
128.266
128.096
128.071
127.863
77.414 cdcl3
77.160 cdcl3
76.905 cdcl3
51.772
26.765
-1.084
f1 (ppm)


O
HO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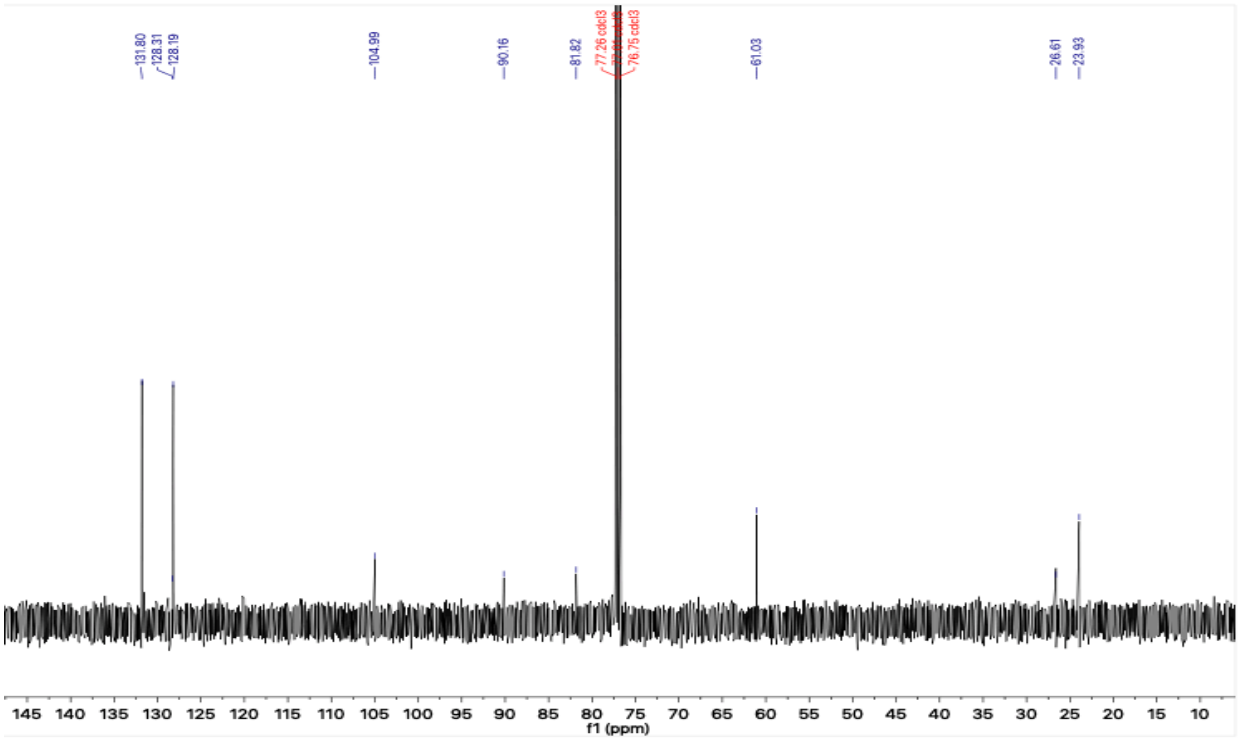
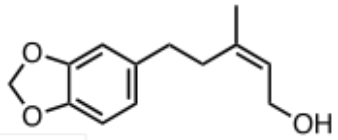
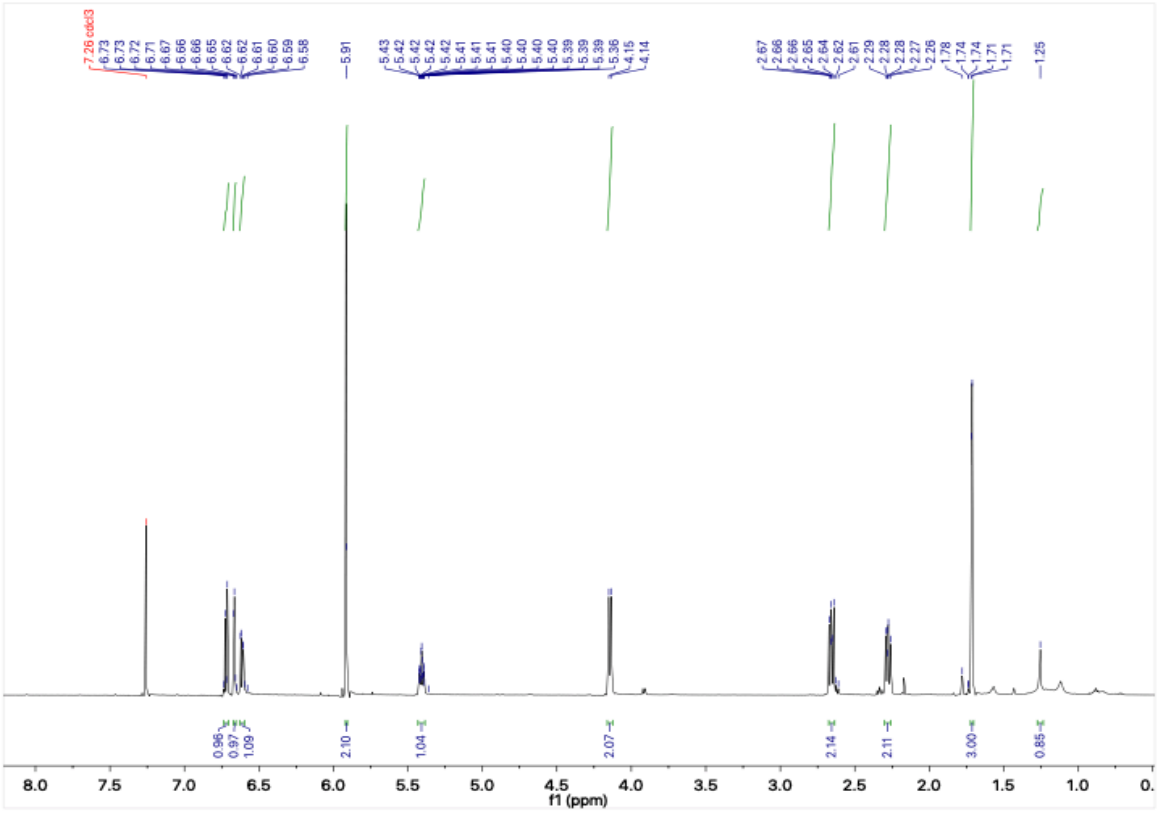
7.88
7.87
7.86
7.86
7.86
7.86
7.48
7.47
7.47
7.46
7.46
3.84
3.83
3.82
3.82
3.81
2.72
2.71
2.70
2.57
1.76
1.75
1.74
2.04
1.99
2.07
2.08
3.00
0.84
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


131.80
128.31
128.19
104.99
90.16
81.82
77.26 cdcl3
76.75 cdcl3
61.03
26.61
23.93
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10
f1 (ppm)


O
O
OH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6.73
6.72
6.71
6.67
6.66
6.66
6.65
6.62
6.62
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6.60
6.59
6.58
5.91
5.43
5.42
5.42
5.42
5.42
5.41
5.41
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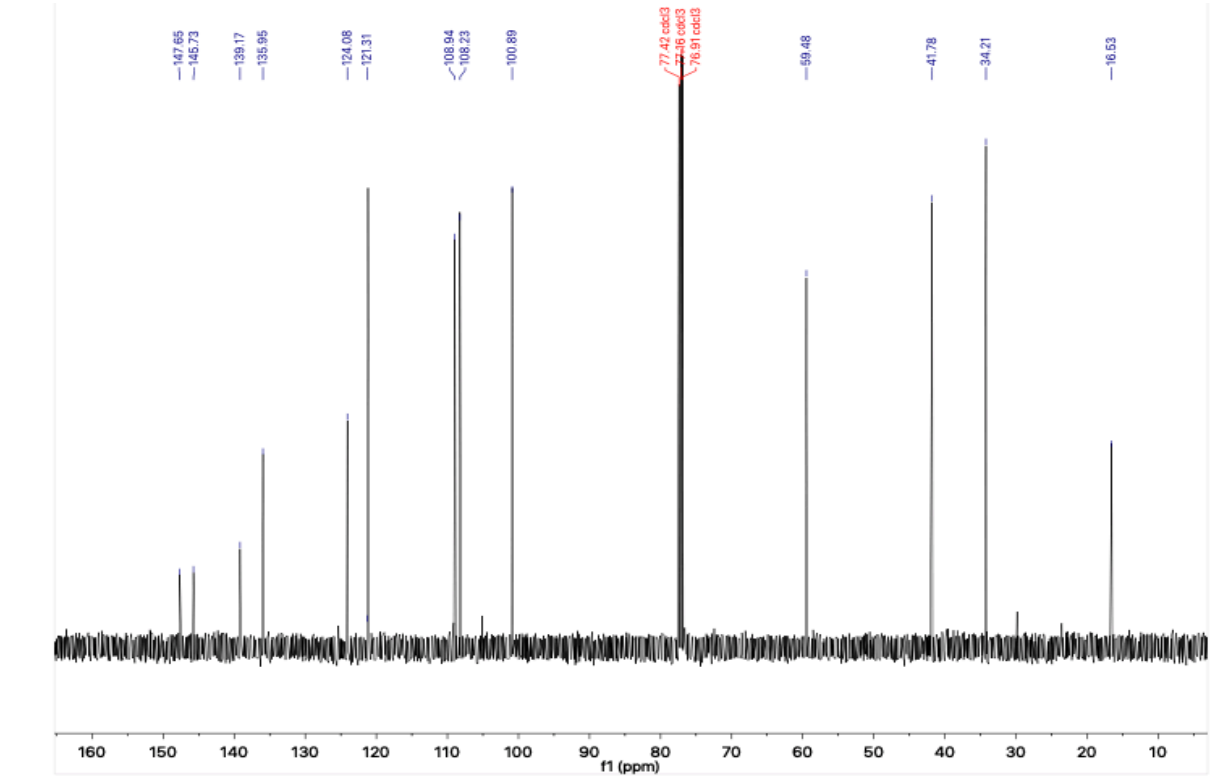
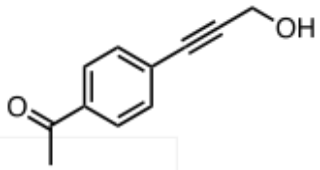
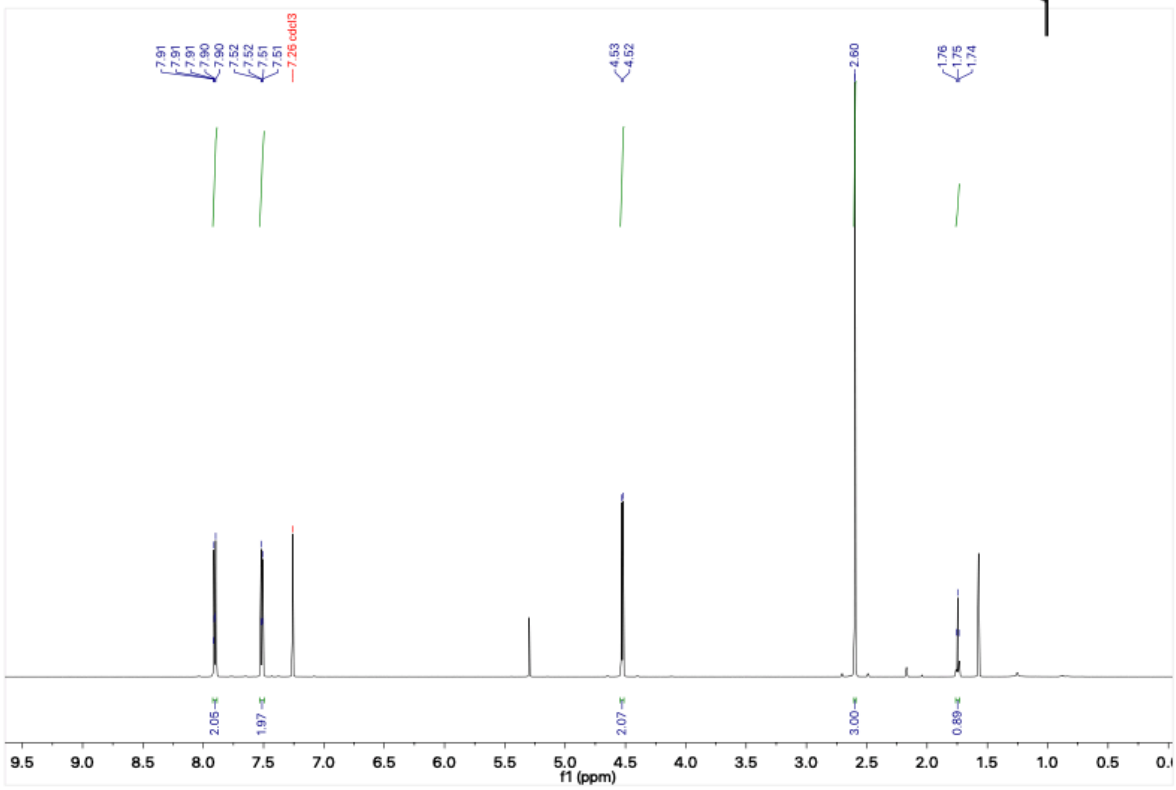
2.61
2.29
2.28
2.28
2.27
2.26
1.78
1.74
1.74
1.71
1.71
1.25
0.96
0.97
1.09
2.10
1.04
2.07
2.14
2.11
3.00
0.85
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)


147.65
145.73
139.17
135.95
124.08
121.31
108.94
108.23
100.89
77.42 cdcl3
77.06 cdcl3
76.91 cdcl3
59.48
41.78
34.21
16.53
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


OH
O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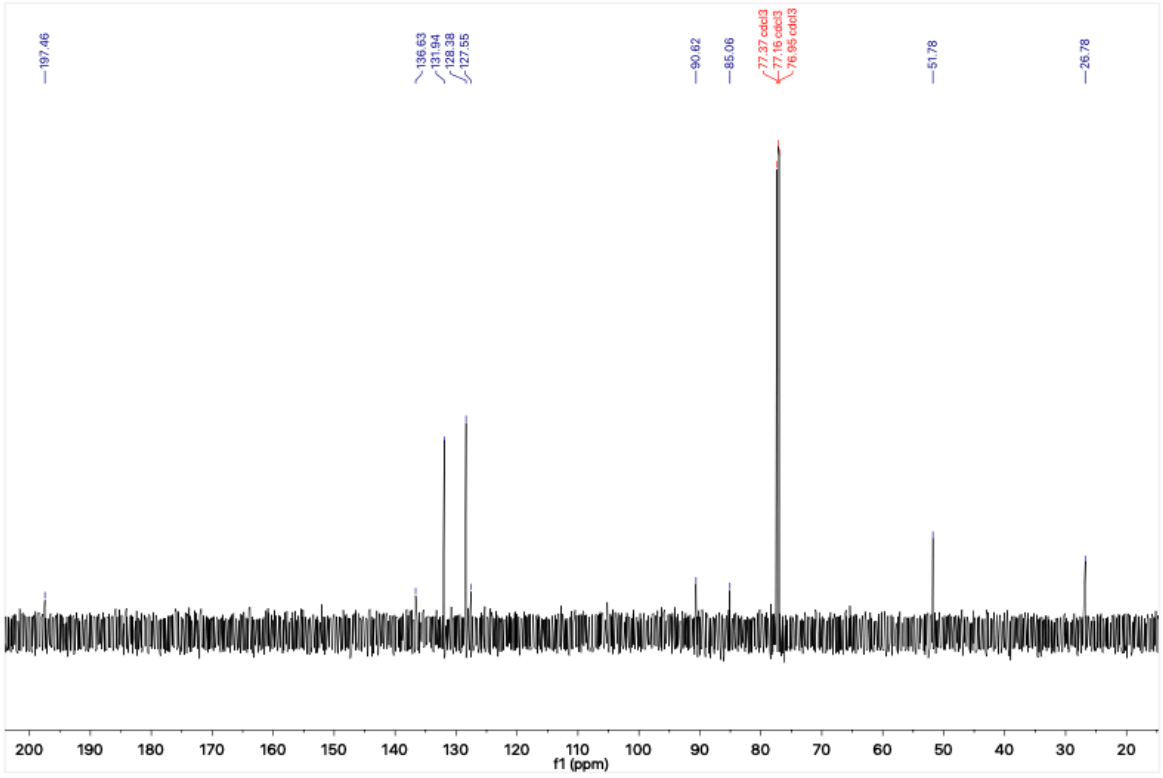
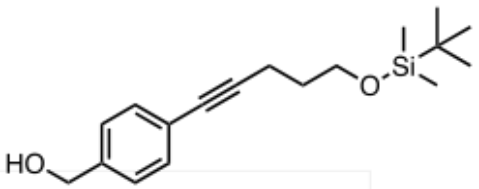
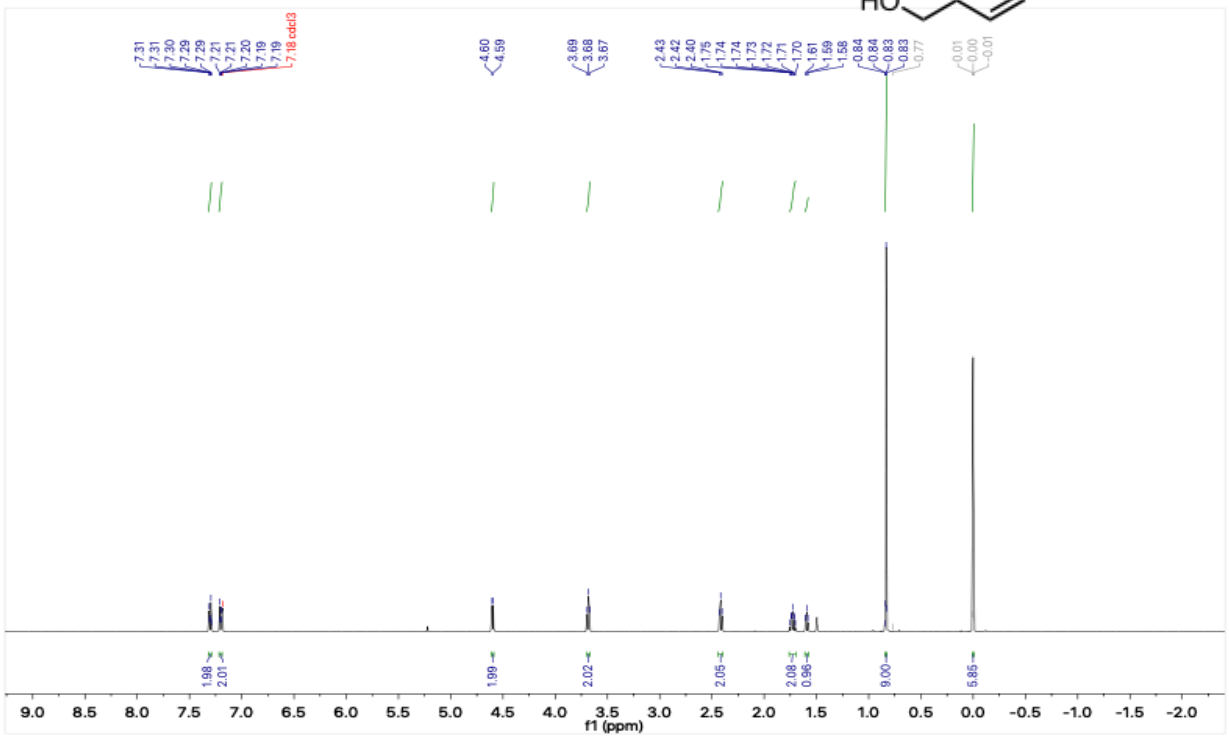
7.91
7.91
7.90
7.90
7.52
7.52
7.51
7.51
7.26 cdcl3
4.53
4.52
2.60
1.76
1.75
1.74
2.05
1.97
2.07
3.00
0.89
f1 (ppm)


197.46
136.63
131.94
128.38
127.55
90.62
85.06
77.37 cdcl3
77.16 cdcl3
76.95 cdcl3
51.78
26.78
f1 (ppm)


HO
O
Si


7.31
7.31
7.30
7.29
7.29
7.21
7.21
7.20
7.19
7.19
7.18 cdcl3
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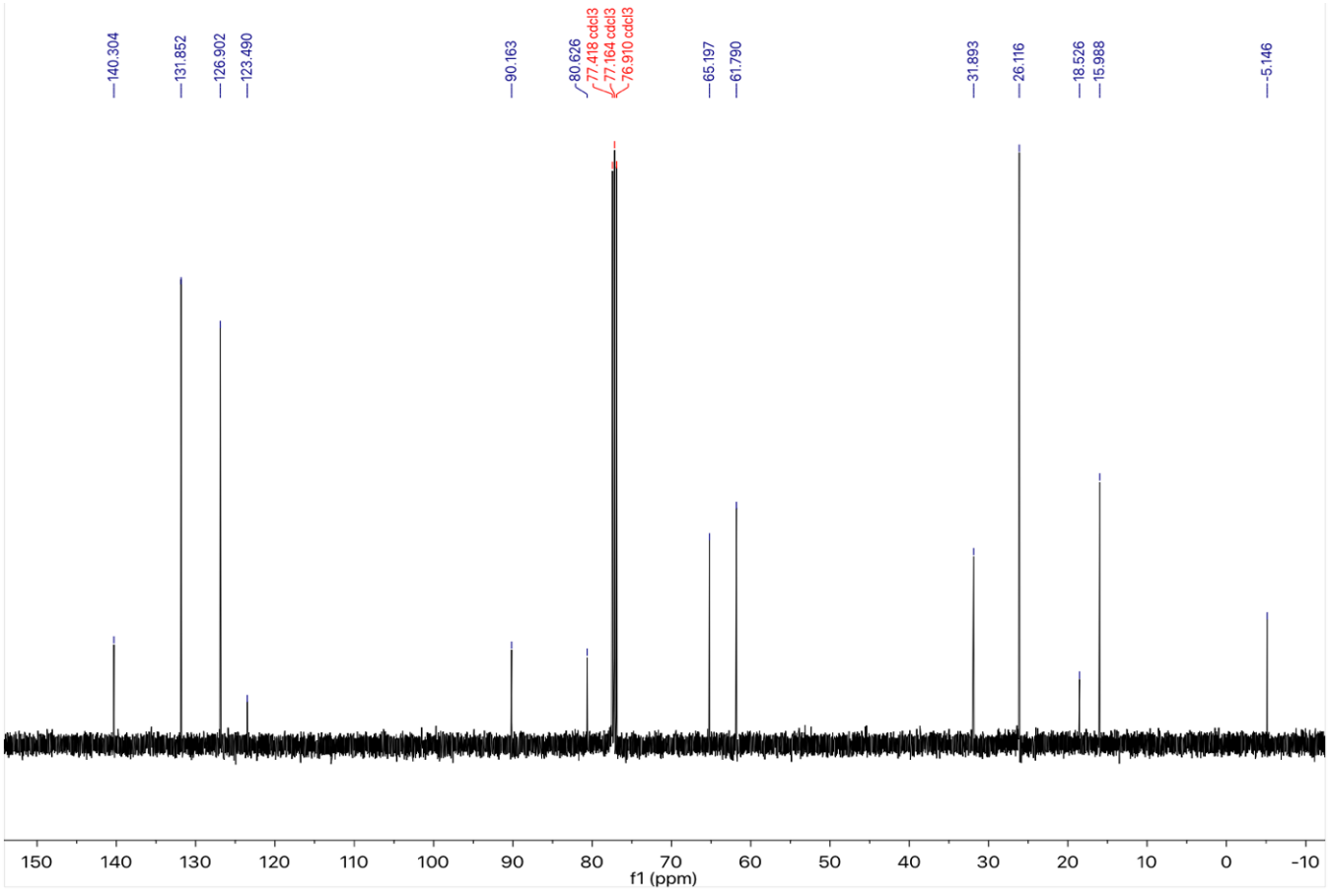
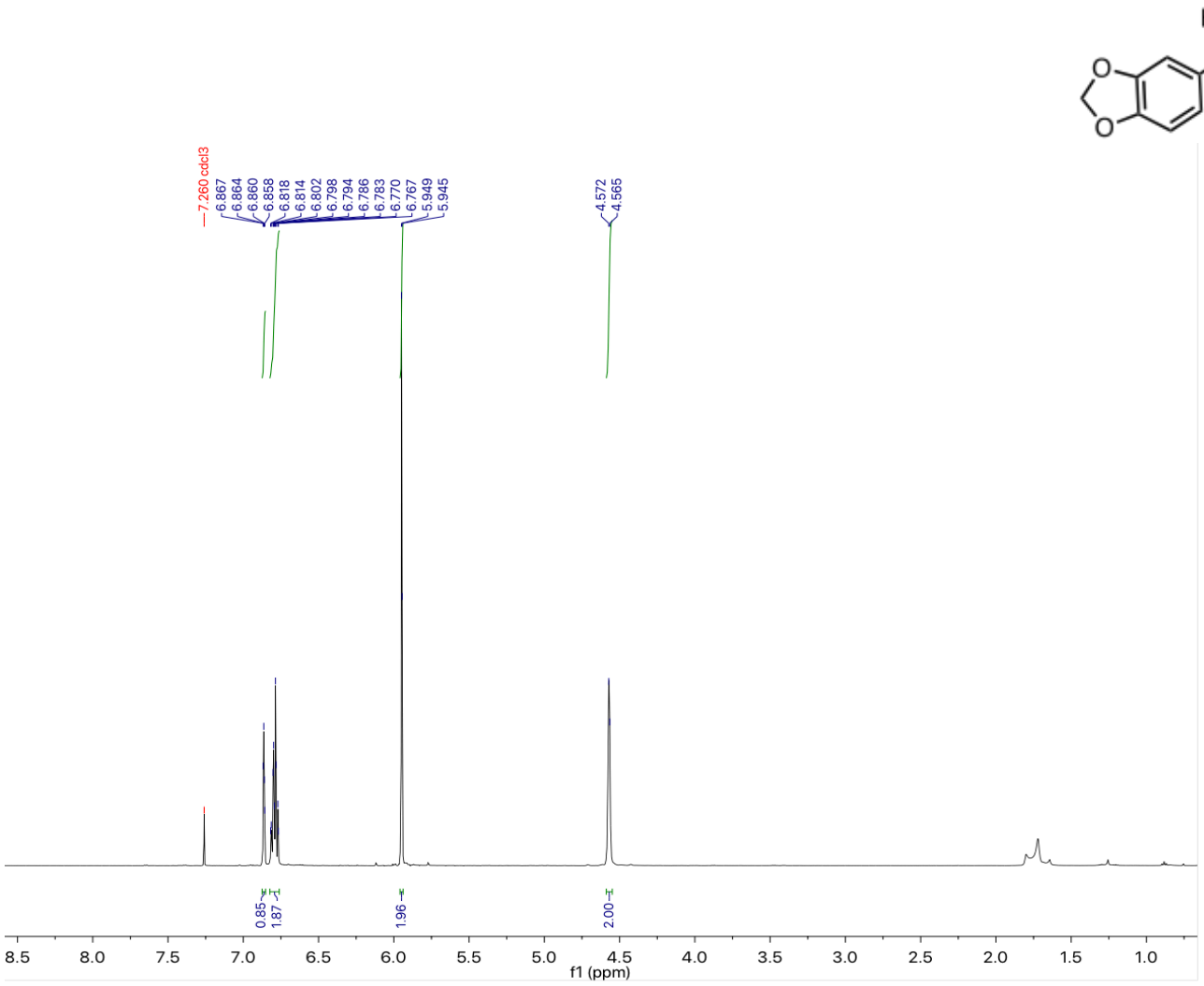
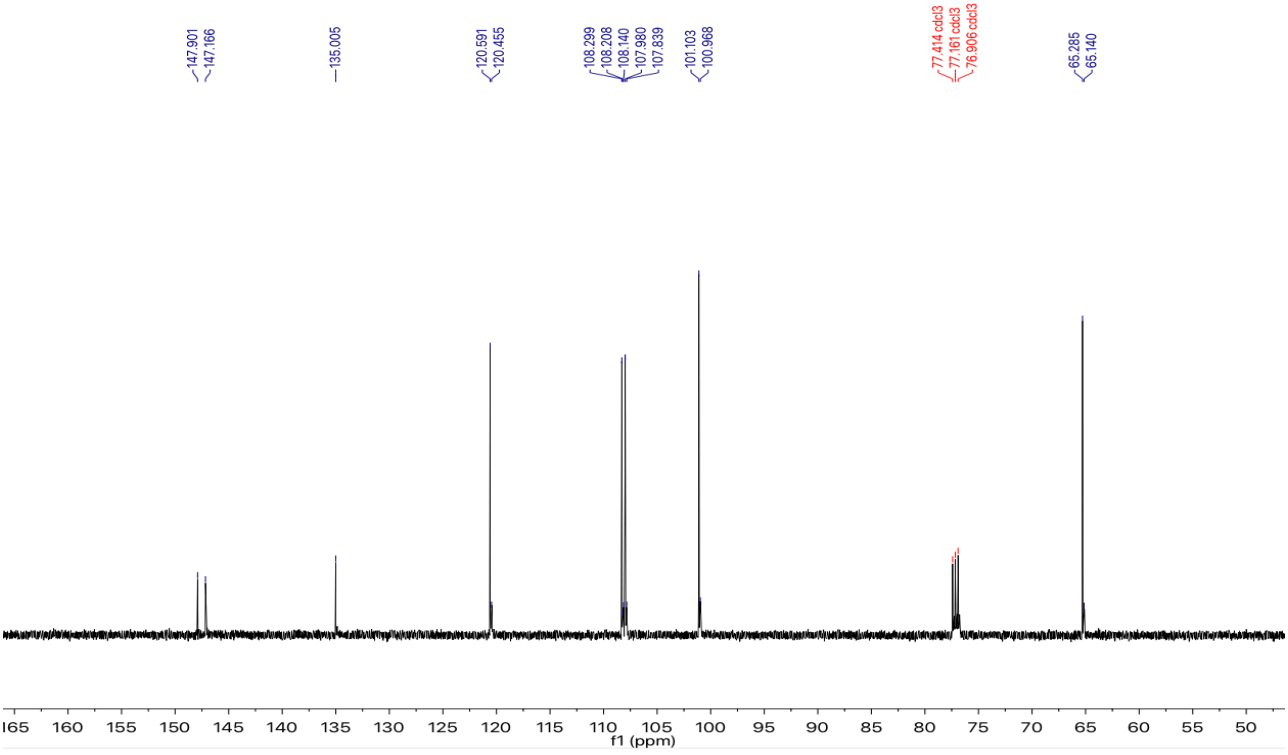
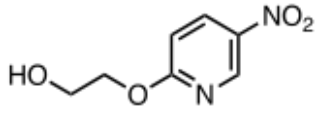
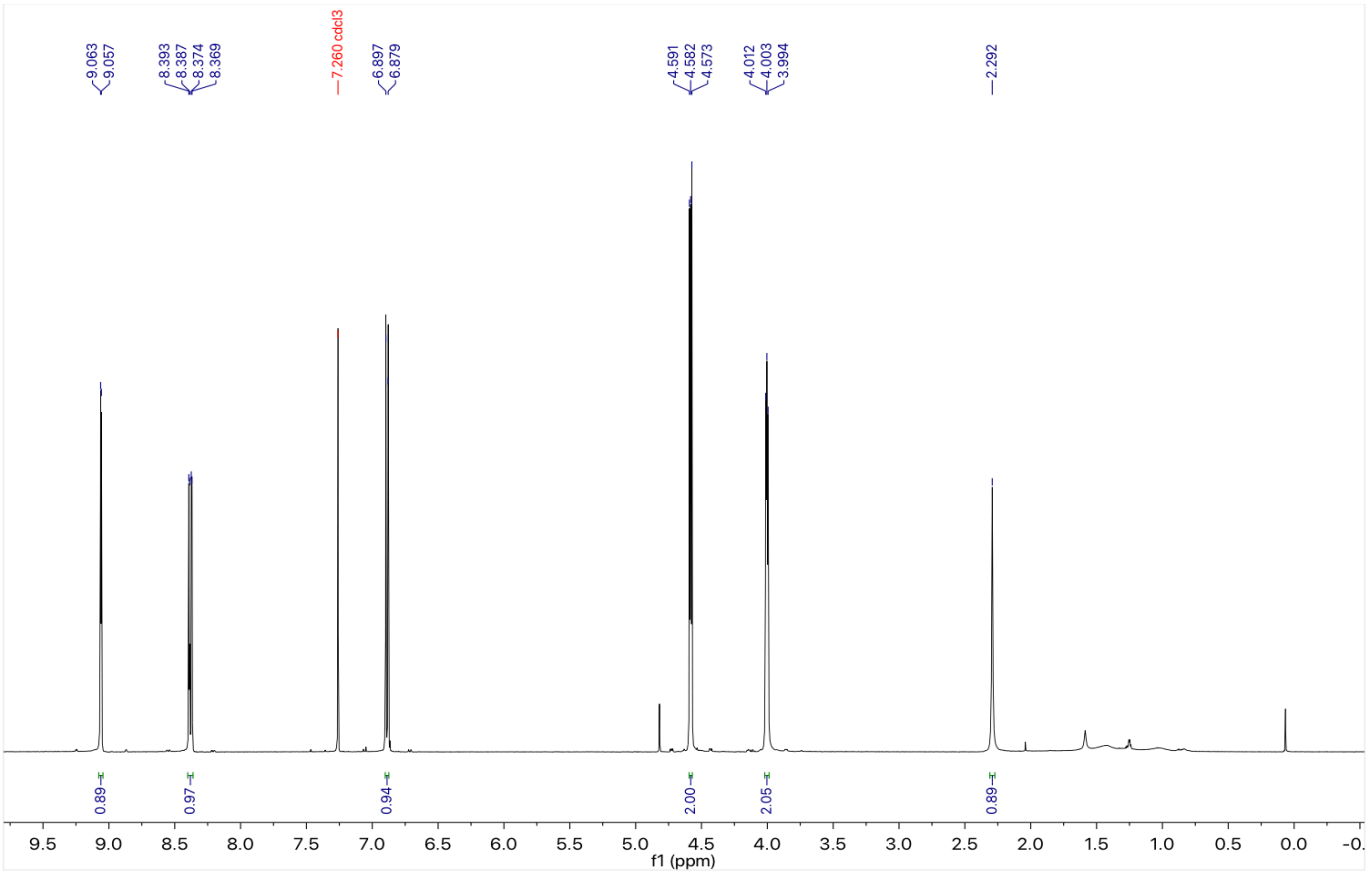
4.59
3.69
3.68
3.67
2.43
2.42
2.40
1.75
1.74
1.74
1.73
1.72
1.71
1.70
1.61
1.59
1.58
0.84
0.84
0.83
0.83
0.77
0.01
0.00
-0.01
1.98
2.01
1.99
2.02
2.05
2.08
0.96
9.00
5.85
f1 (ppm)

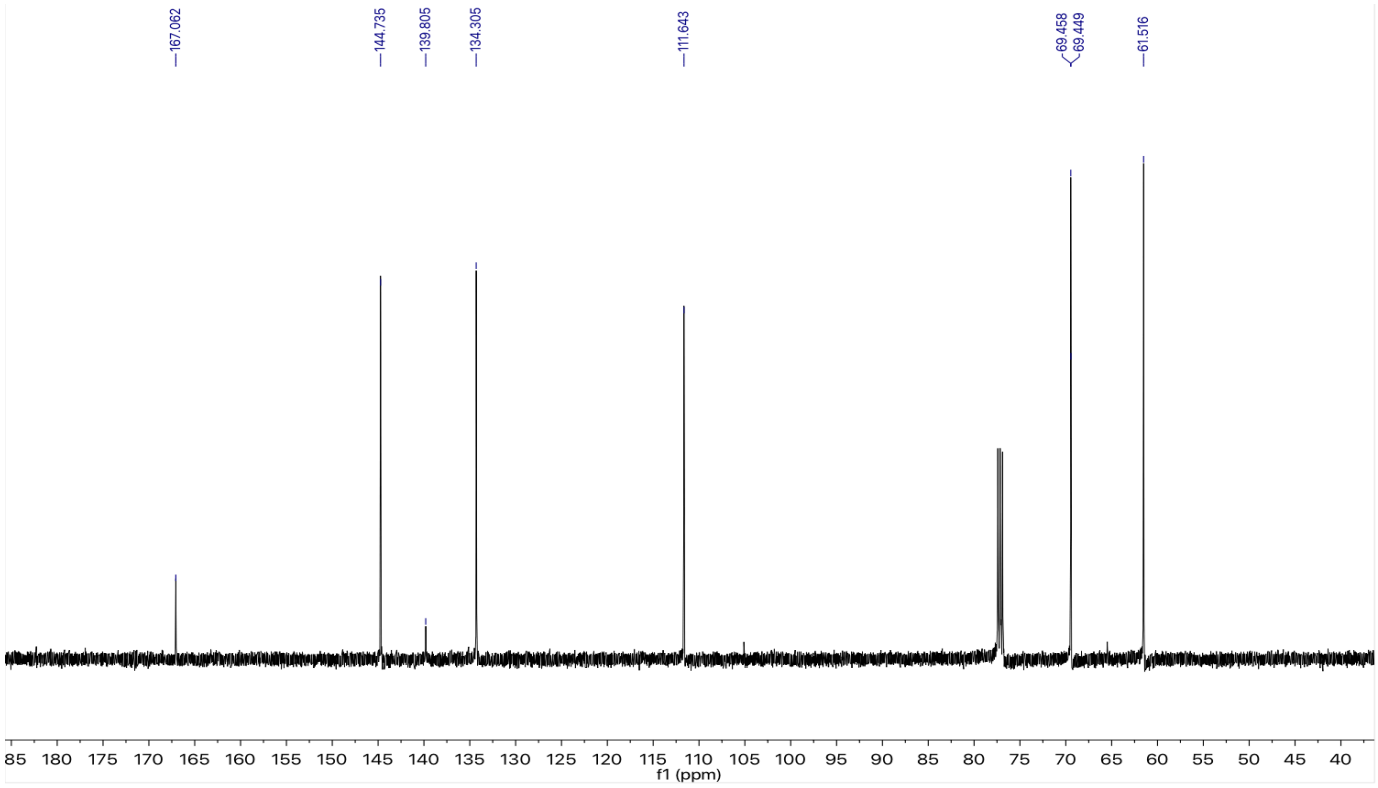

140.304
131.852
126.902
123.490
90.163
80.626
77.418 cdcl3
77.164 cdcl3
76.910 cdcl3
65.197
61.790
31.893
26.116
18.526
15.988
-5.146
f1 (ppm)

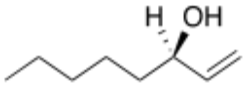

HO
7.260 cdcl3
6.867
6.864
6.860
6.858
6.818
6.814
6.802
6.798
6.794
6.786
6.783
6.770
6.767
5.949
5.945
4.572
4.565
0.85
1.87
1.96
2.00
f1 (ppm)

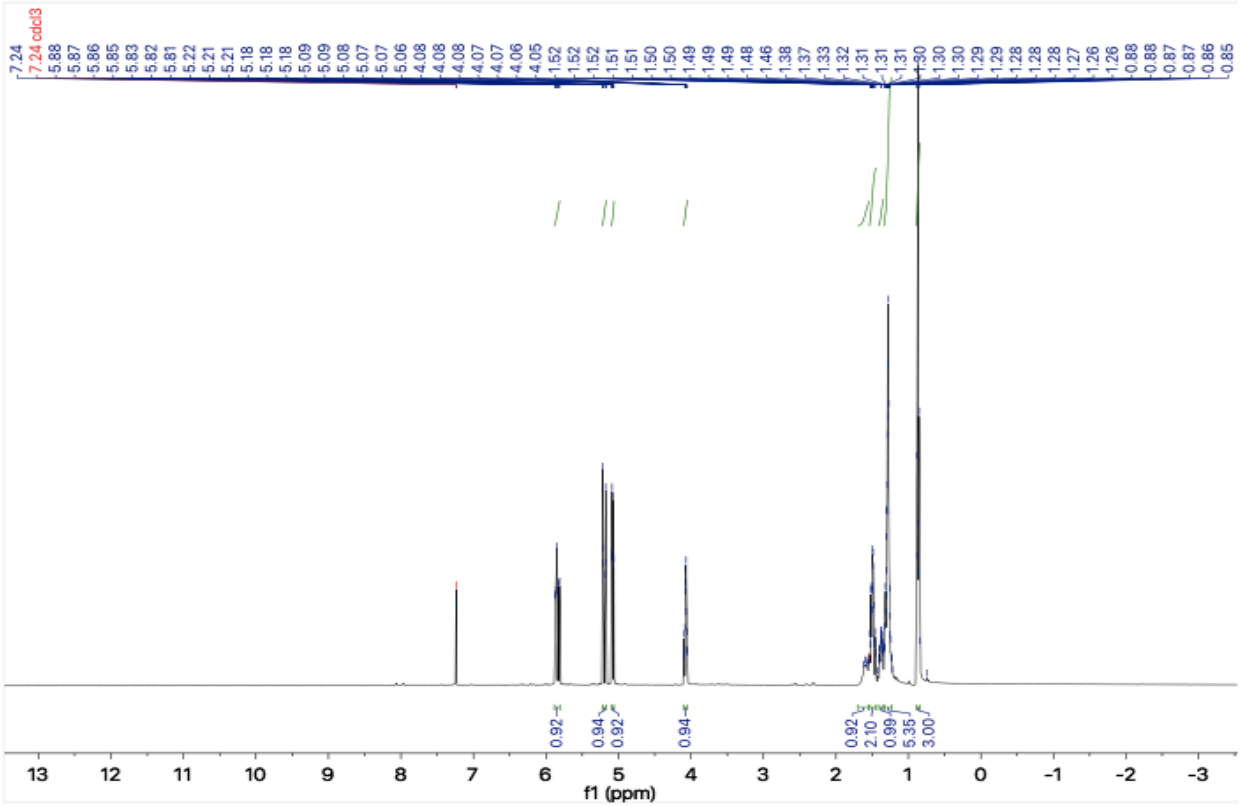

147.901
147.166
135.005
120.591
120.455
108.299
108.208
108.140
107.980
107.839
101.103
100.968
77.414 cdcl3
77.161 cdcl3
76.906 cdcl3
65.285
65.140
f1 (ppm)

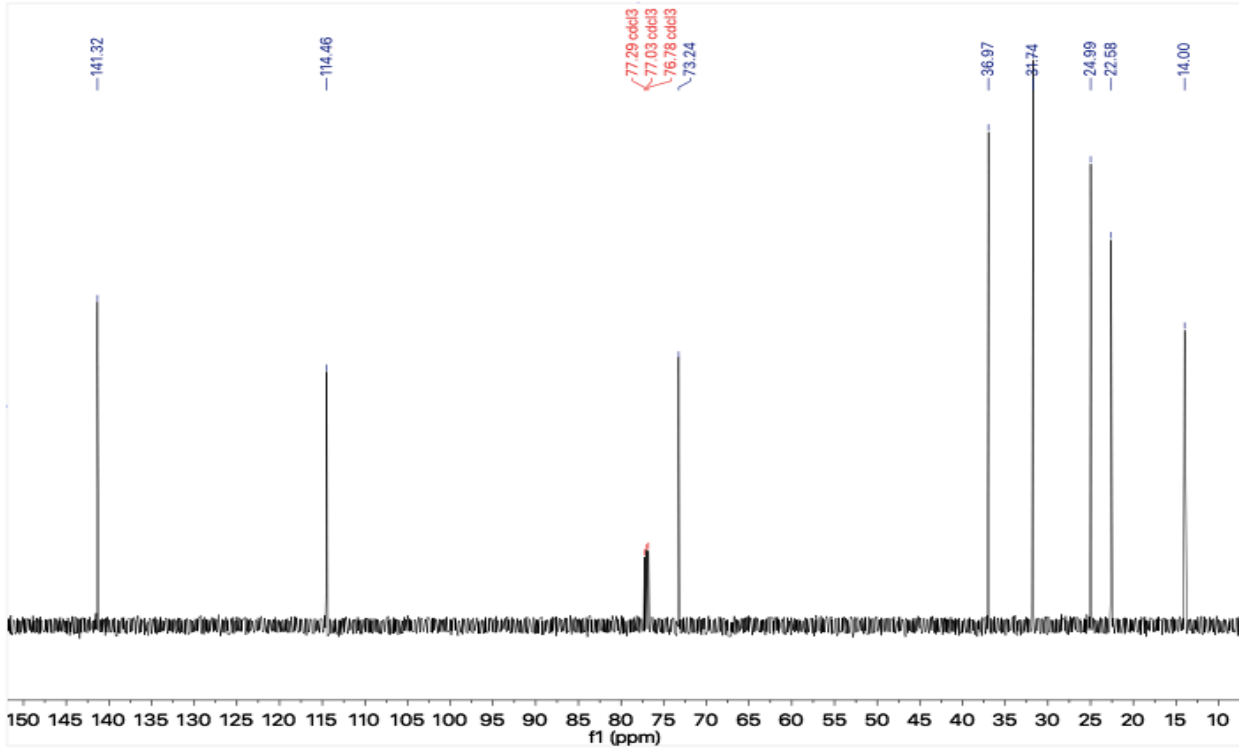

NO2
HO
O
N

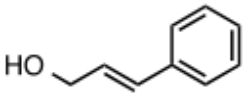

9.063
9.057
8.393
8.387
8.374
8.369
7.260 cdcl3
6.897
6.879
4.591
4.582
4.573
4.012
4.003
3.994
2.292
0.89
0.97
0.94
2.00
2.05
0.89
f1 (ppm)

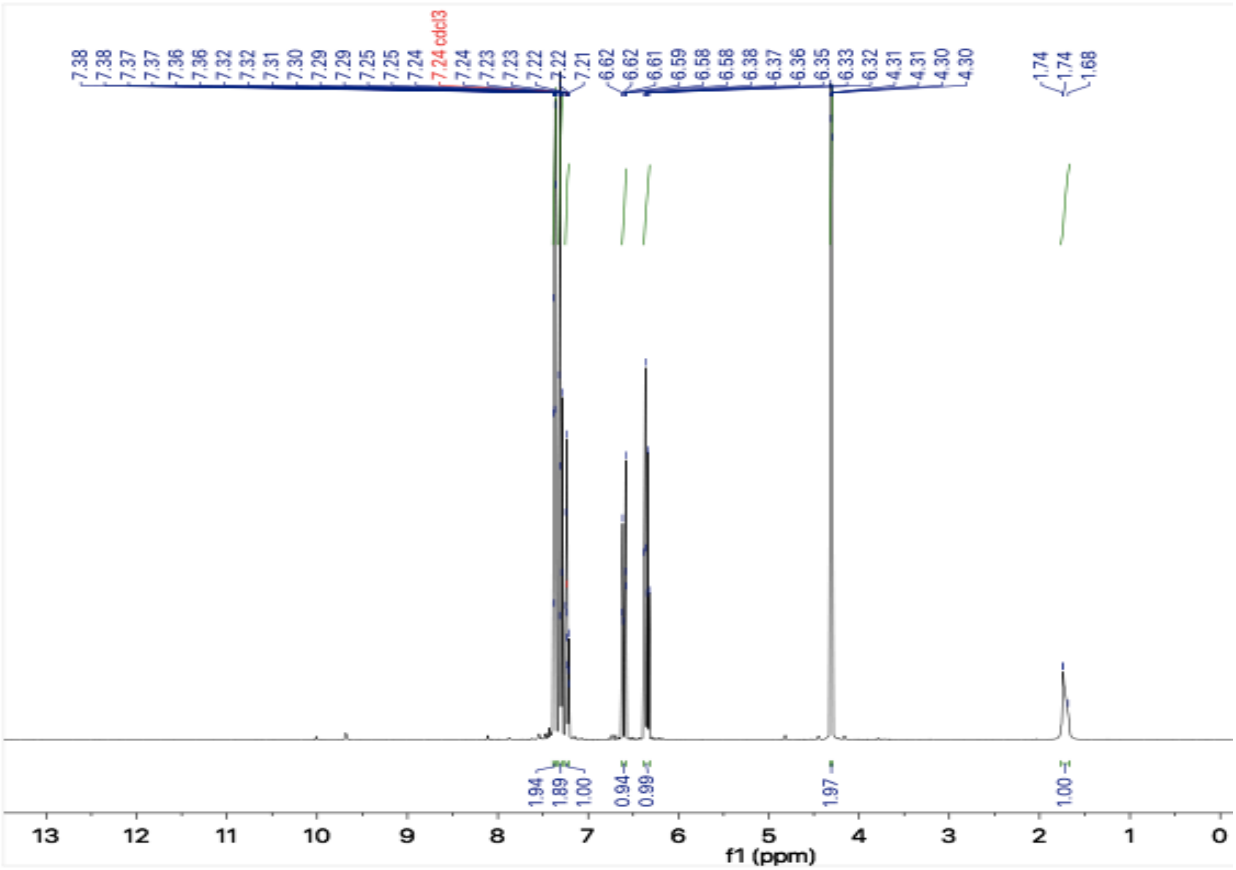

167.062
144.735
139.805
134.305
111.643
69.458
69.449
61.516
f1 (ppm)

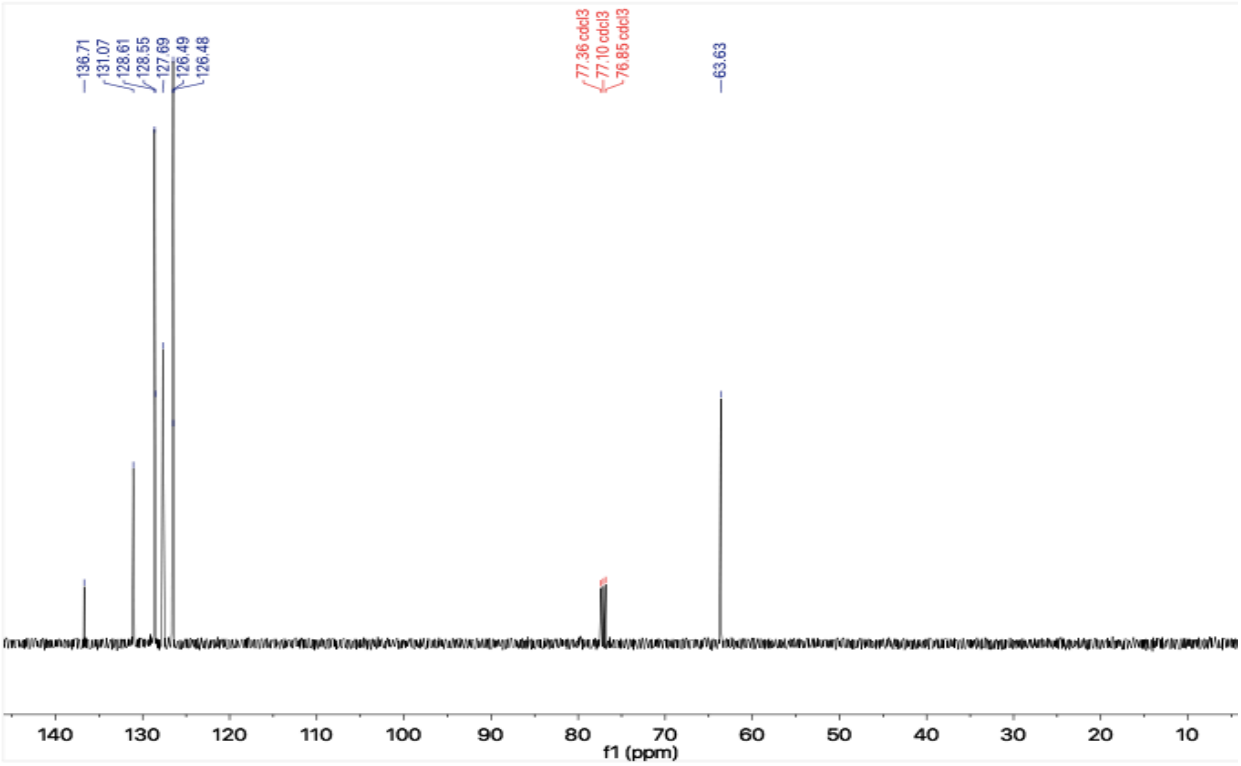

H
OH

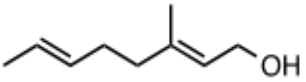

7.24
7.24 cdcl3
5.88
5.87
5.86
5.85
5.83
5.82
5.81
5.22
5.21
5.21
5.18
5.18
5.18
5.09
5.09
5.08
5.07
5.07
5.06
4.08
4.08
4.08
4.07
4.07
4.06
4.05
1.52
1.52
1.52
1.51
1.51
1.50
1.50
1.49
1.49
1.49
1.48
1.46
1.38
1.37
1.33
1.32
1.31
1.31
1.31
1.30
1.30
1.30
1.29
1.29
1.28
1.28
1.28
1.27
1.26
1.26
0.88
0.88
0.87
0.87
0.86
0.85
0.92
0.94
0.92
0.94
0.92
2.10
0.99
5.35
3.00
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

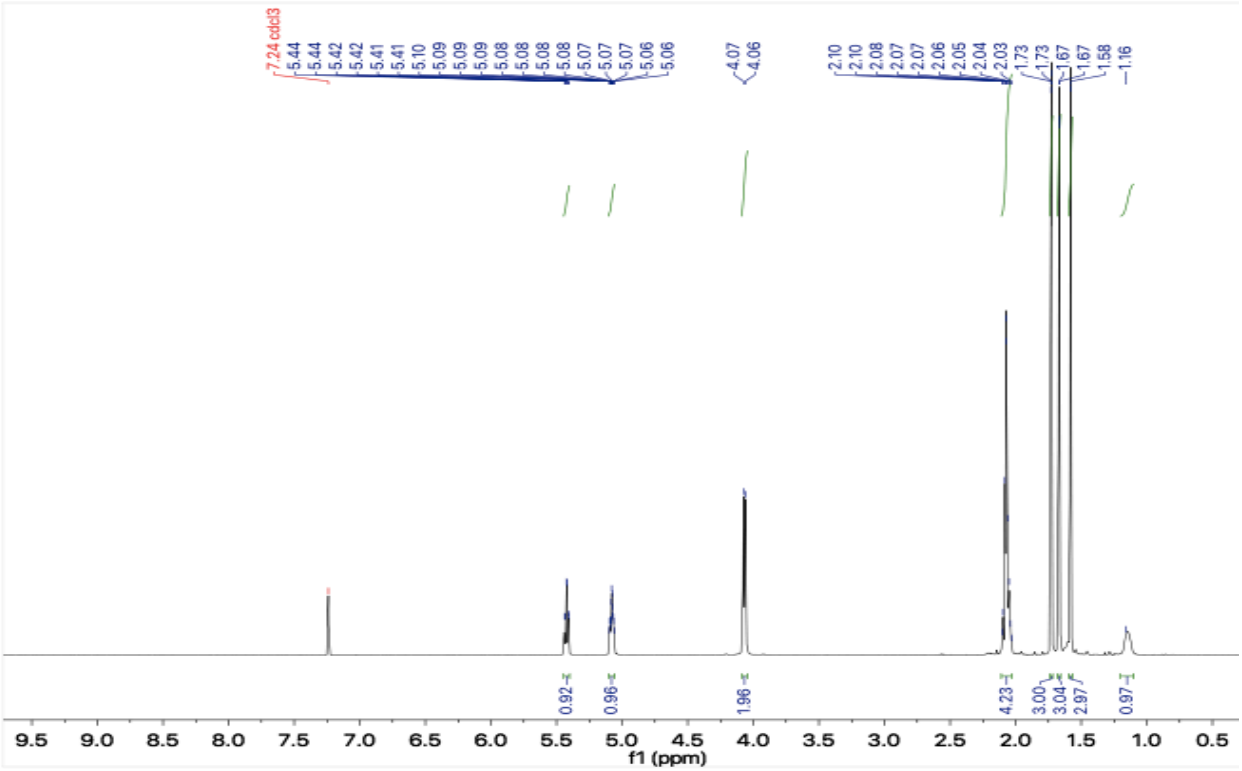

141.32
114.46
77.29 cdcl3
77.03 cdcl3
76.78 cdcl3
73.24
36.97
31.74
24.99
22.58
14.00
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10
f1 (ppm)

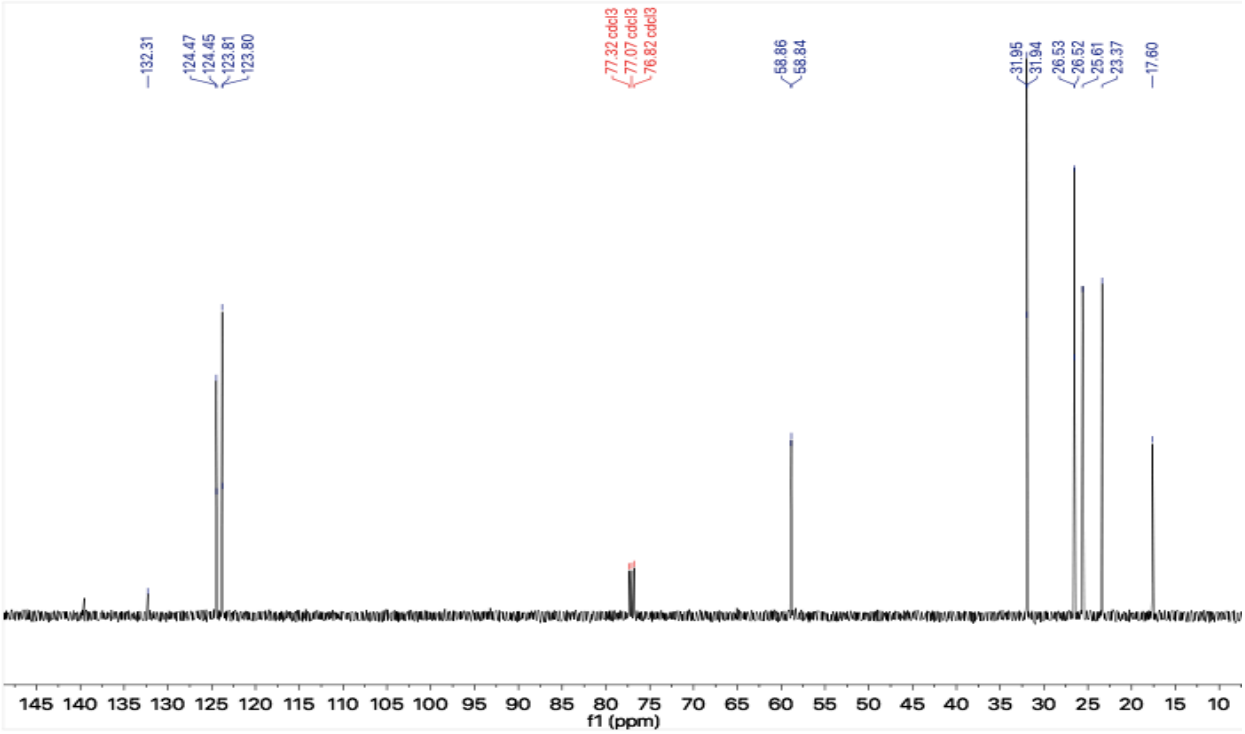

HO

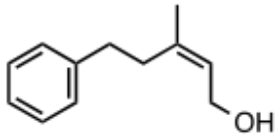

7.38
7.38
7.37
7.37
7.36
7.36
7.32
7.32
7.31
7.30
7.29
7.29
7.25
7.25
7.24
7.24 cdcl3
7.24
7.23
7.23
7.22
7.22
7.21
6.62
6.62
6.61
6.59
6.58
6.58
6.38
6.37
6.36
6.35
6.33
6.32
4.31
4.31
4.30
4.30
1.74
1.74
1.68
1.94
1.89
1.00
0.94
0.99
1.97
1.00
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

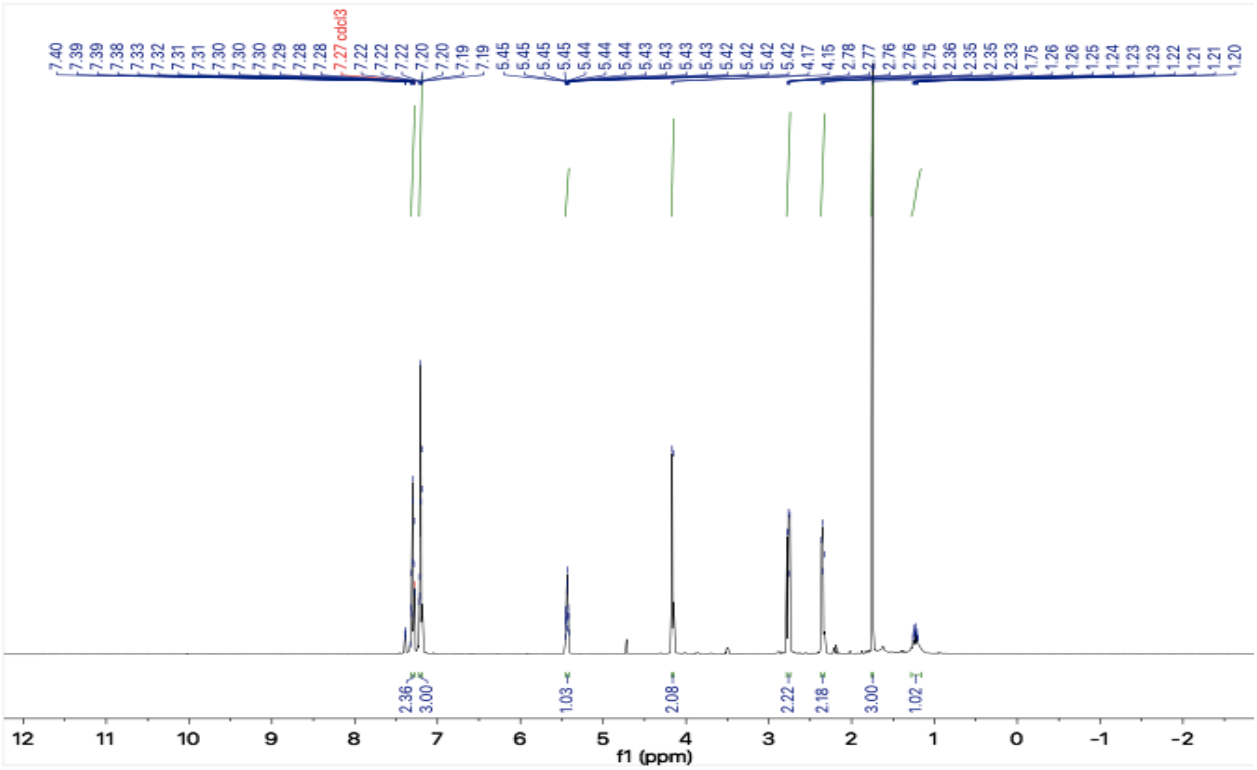

136.71
131.07
128.61
128.55
127.69
126.49
126.48
77.36 cdcl3
77.10 cdcl3
76.85 cdcl3
63.63
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

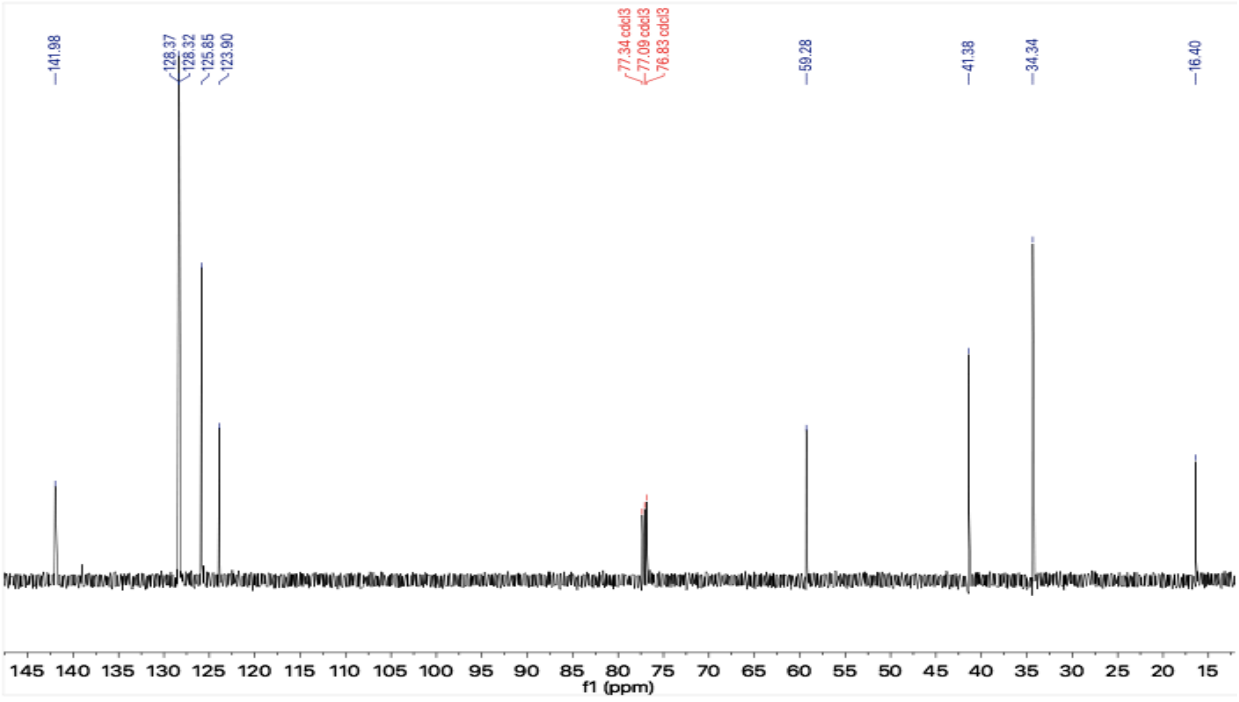

OH

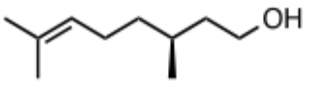

7.24 cdcl3
5.44
5.44
5.42
5.42
5.41
5.41
5.10
5.09
5.09
5.09
5.08
5.08
5.08
5.07
5.07
5.07
5.06
5.06
4.07
4.06
2.10
2.10
2.08
2.07
2.07
2.06
2.05
2.04
2.03
1.73
1.73
1.67
1.67
1.58
1.16
0.92
0.96
1.96
4.23
3.00
3.04
2.97
0.97
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

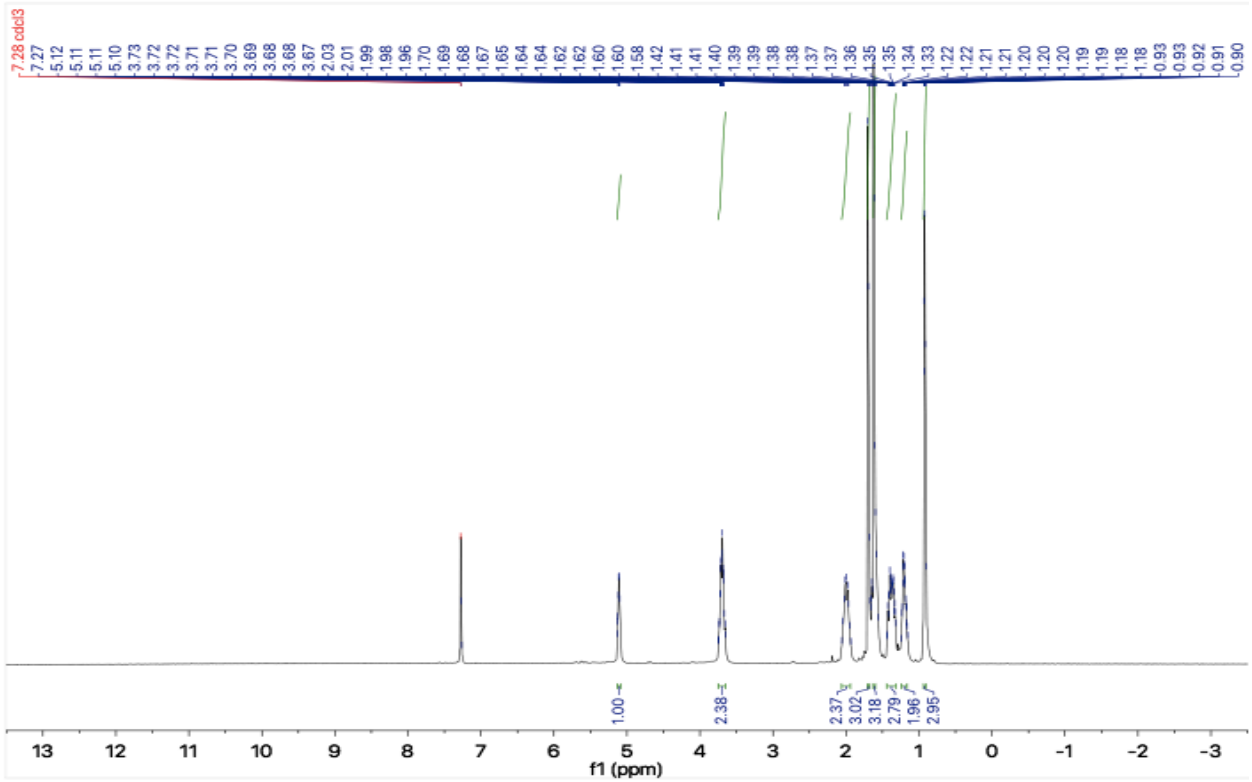

132.31
124.47
124.45
123.81
123.80
77.32 cdcl3
77.07 cdcl3
76.82 cdcl3
58.86
58.84
31.95
31.94
26.53
26.52
25.61
23.37
17.60
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10
f1 (ppm)


OH


7.40 7.39 7.39 7.38 7.33 7.32 7.31 7.31 7.30 7.30 7.30 7.29 7.28 7.28 7.27 cdcl3 7.22 7.22 7.22 7.20 7.20 7.19 7.19
5.45 5.45 5.45 5.44 5.44 5.44 5.43 5.43 5.43 5.43 5.42 5.42 5.42 5.42
4.17 4.15
2.78 2.77 2.76 2.76 2.75 2.36 2.35 2.35 2.33 1.75 1.26 1.26 1.25 1.24 1.23 1.23 1.22 1.21 1.21 1.20
2.36 3.00 1.03 2.08 2.22 2.18 3.00 1.02
12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2
f1 (ppm)


141.98
128.37 128.32 125.85 123.90
77.34 cdcl3 77.09 cdcl3 76.83 cdcl3
69.28
41.38
34.34
16.40
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15
f1 (ppm)


OH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7.27
5.12
5.11
5.11
5.10
3.73
3.72
3.72
3.71
3.71
3.70
3.69
3.68
3.68
3.67
2.03
2.01
1.99
1.98
1.96
1.70
1.69
1.68
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1.65
1.64
1.64
1.62
1.62
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1.60
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1.39
1.39
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1.38
1.37
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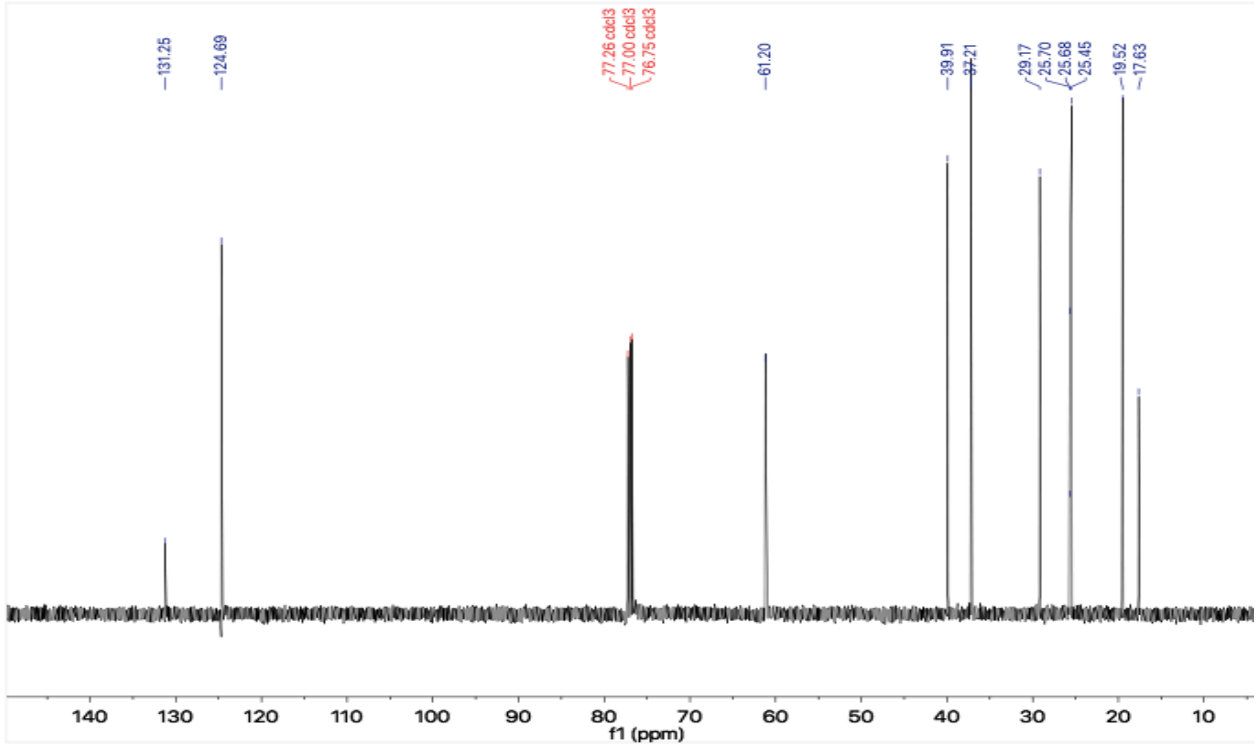
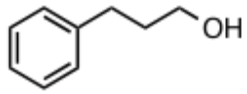
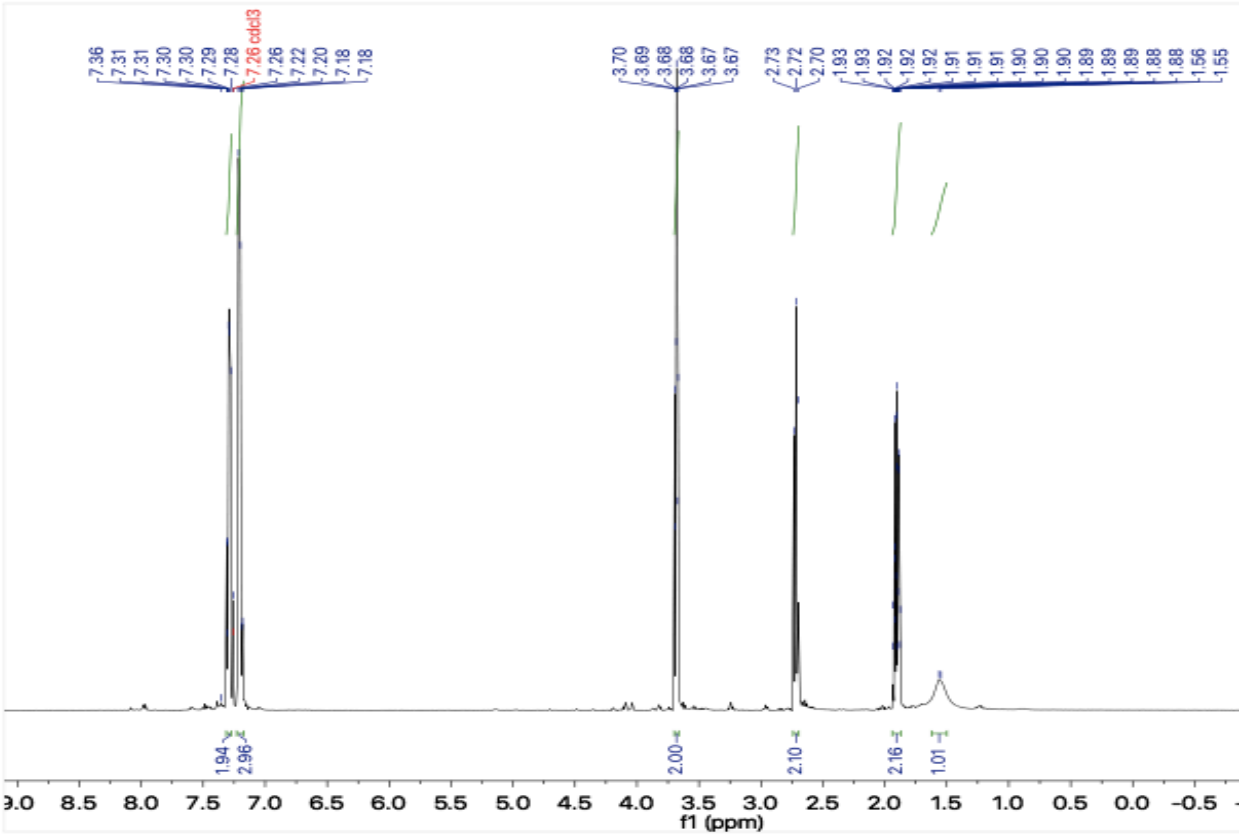
1.34
1.33
1.22
1.22
1.21
1.21
1.20
1.20
1.20
1.19
1.19
1.18
1.18
0.93
0.93
0.92
0.91
0.90
1.00
2.38
2.37
3.02
3.18
2.79
1.96
2.95
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)


131.25
124.69
77.26 cdcl3
77.00 cdcl3
76.75 cdcl3
61.20
39.91
37.21
29.17
25.70
25.68
25.45
19.52
17.63
140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


OH


7.36
7.31
7.31
7.30
7.30
7.29
7.28
7.26 cdcl3
7.26
7.22
7.20
7.18
7.18
3.70
3.69
3.68
3.68
3.67
3.67
2.73
2.72
2.70
1.93
1.93
1.92
1.92
1.92
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1.91
1.91
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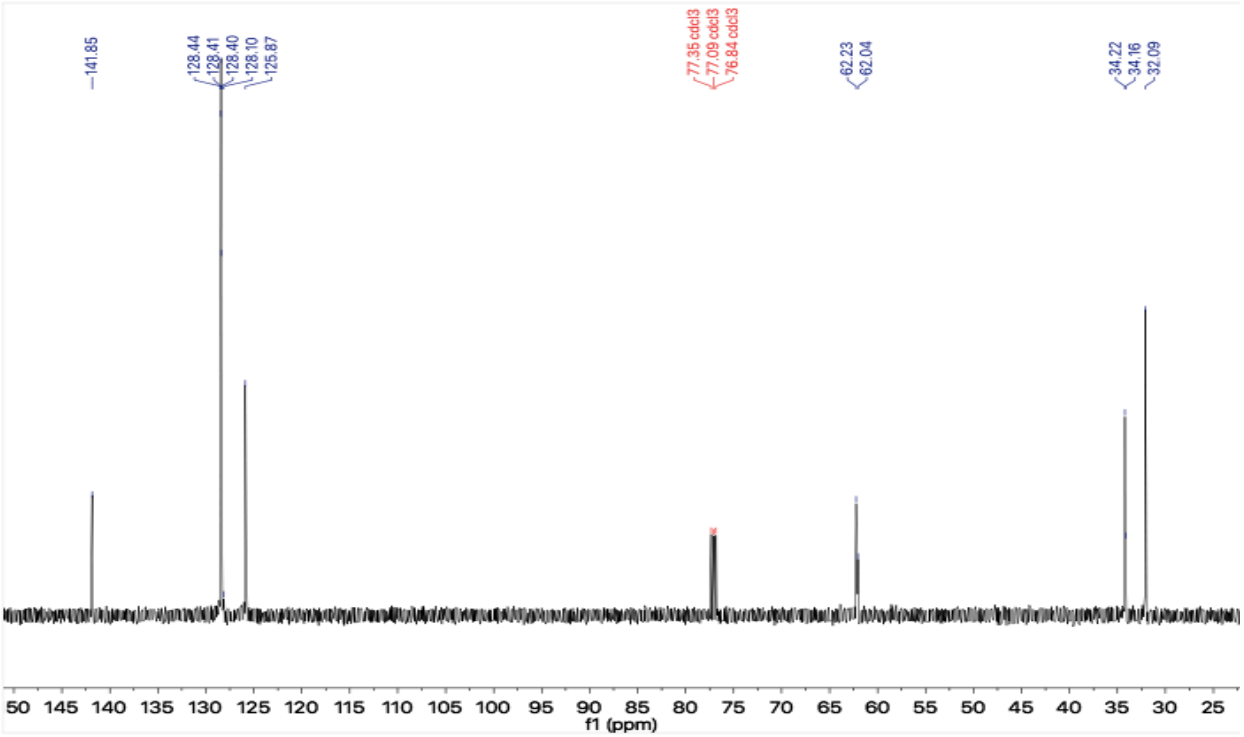
2.00
2.10
2.16
1.01
f1 (ppm)


141.85
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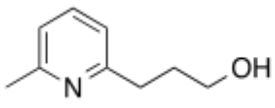
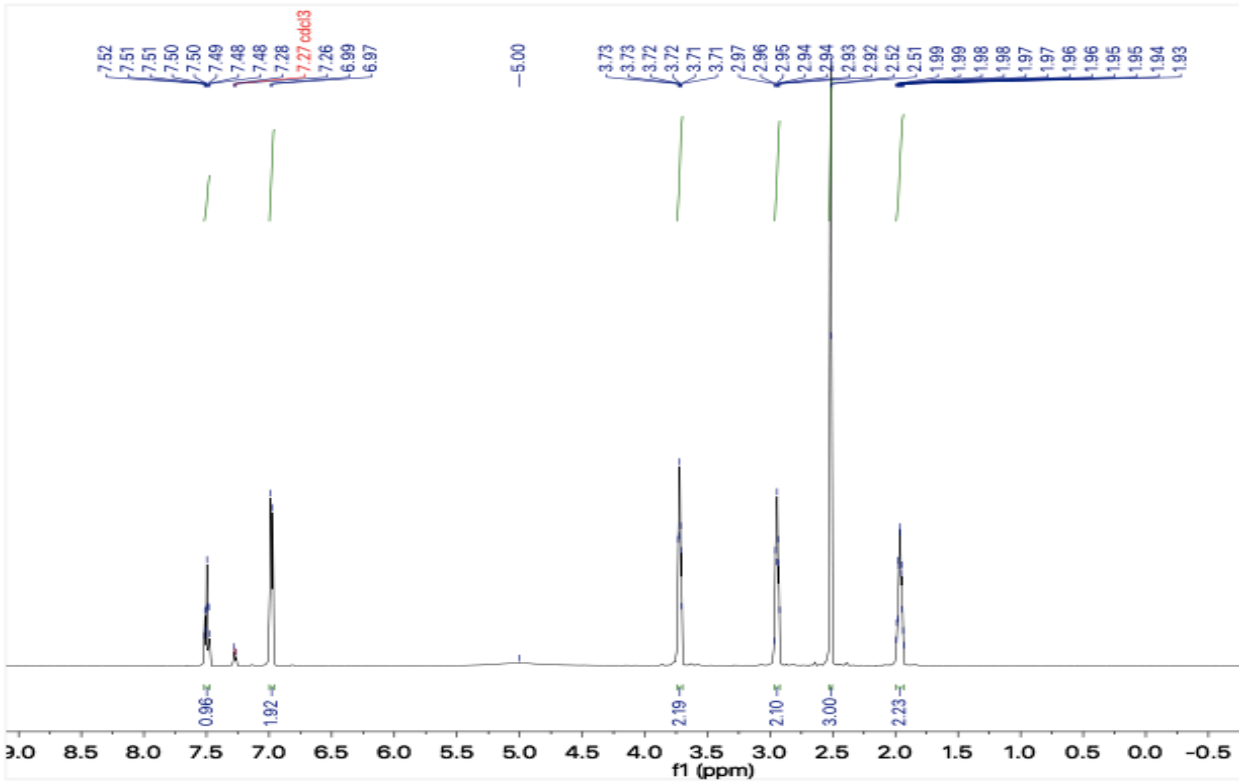
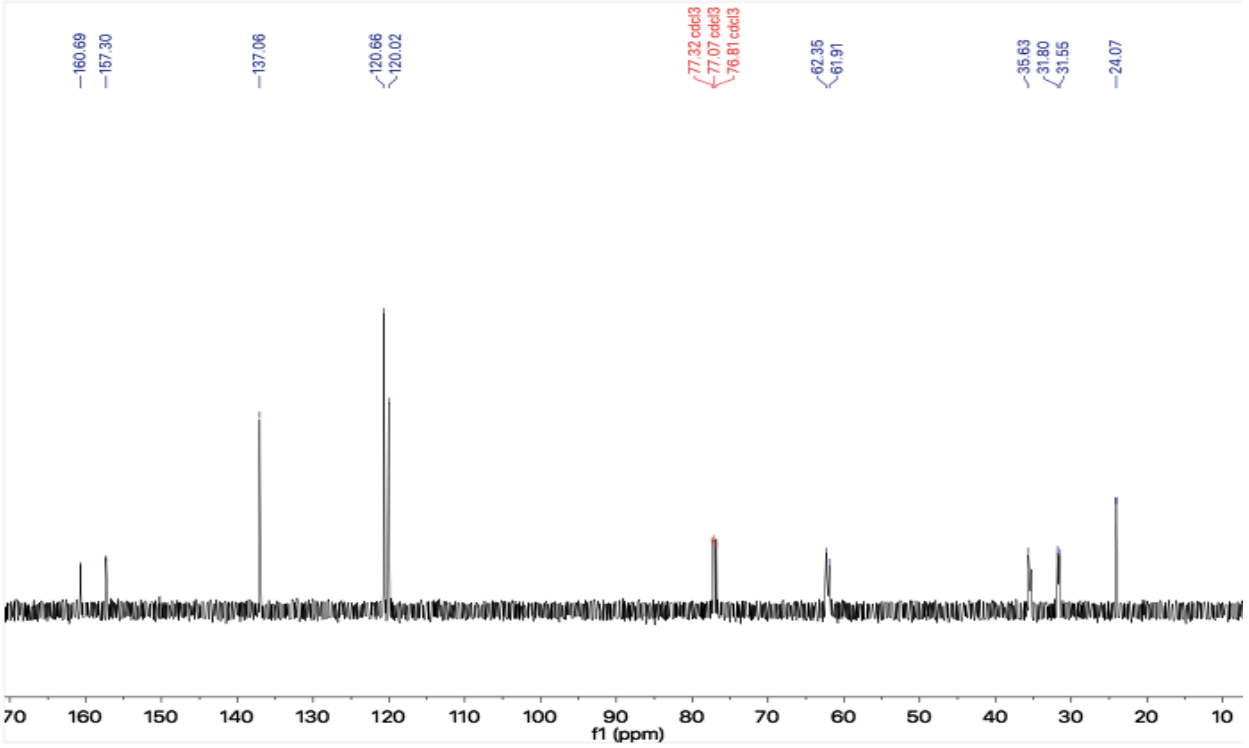
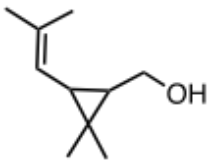
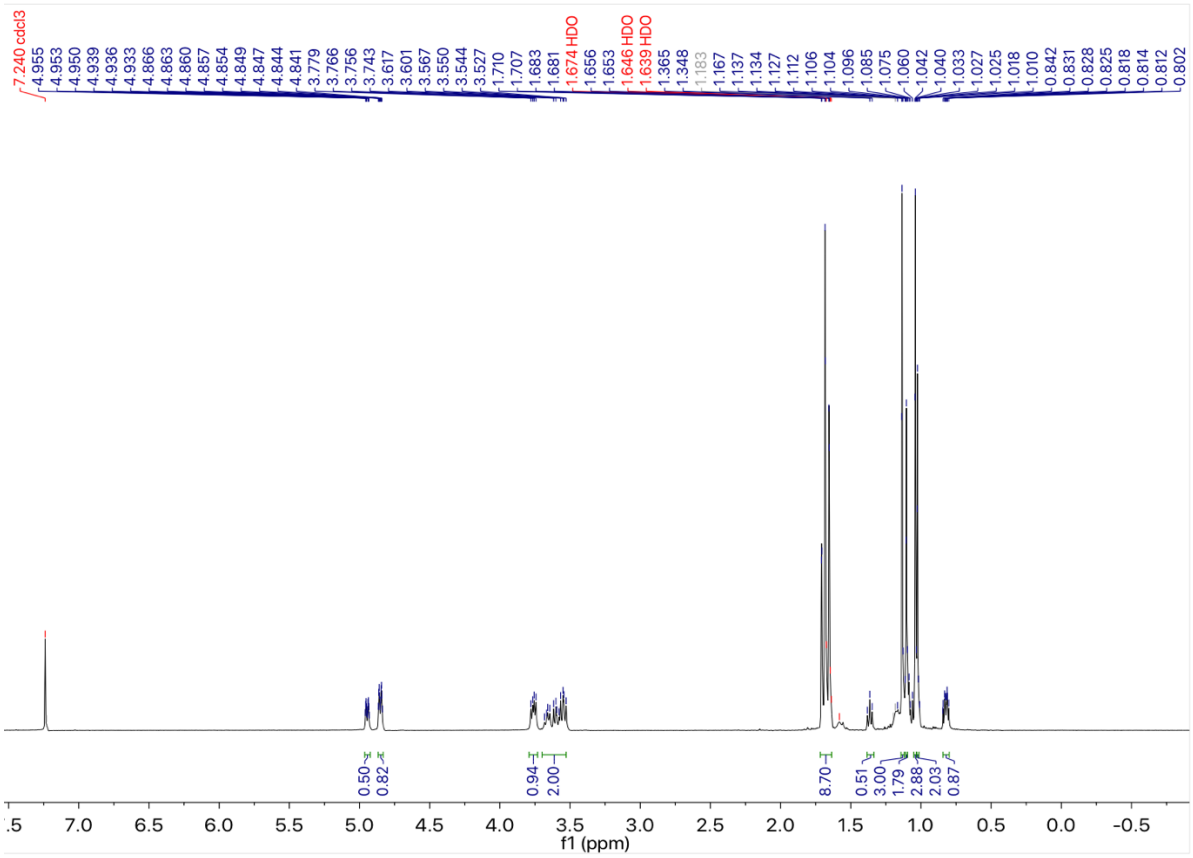
128.40
128.10
125.87
77.35 cdcl3
77.09 cdcl3
76.84 cdcl3
62.23
62.04
34.22
34.16
32.09
f1 (ppm)

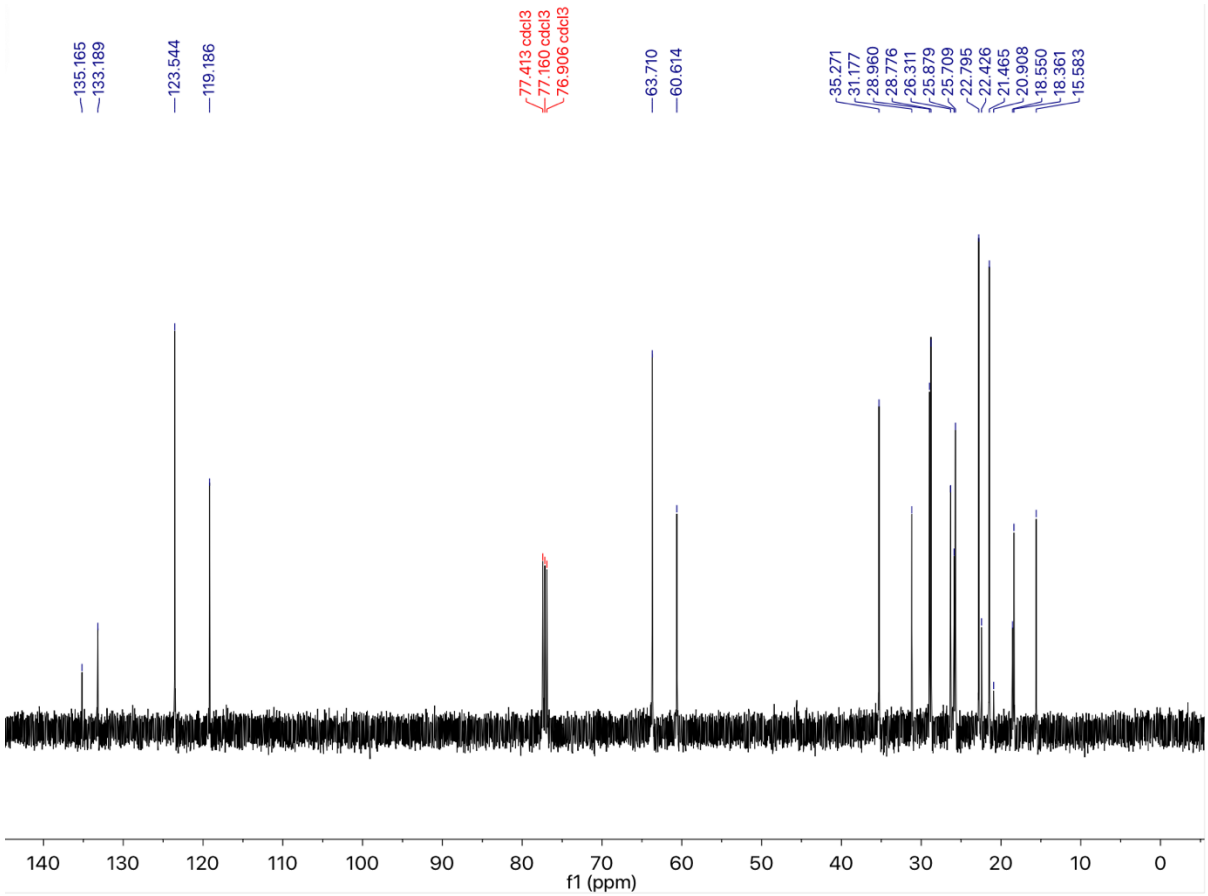

OH
N

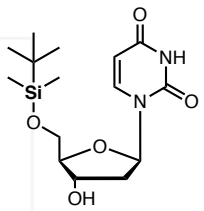

7.52
7.51
7.51
7.50
7.50
7.49
7.48
7.48
7.28
7.27 cdcl3
7.26
6.99
6.97
5.00
3.73
3.73
3.72
3.72
3.71
3.71
2.97
2.96
2.95
2.94
2.94
2.93
2.92
2.52
2.51
1.99
1.99
1.98
1.98
1.97
1.97
1.96
1.96
1.95
1.95
1.94
1.93
0.96
1.92
2.19
2.10
3.00
2.23
f1 (ppm)

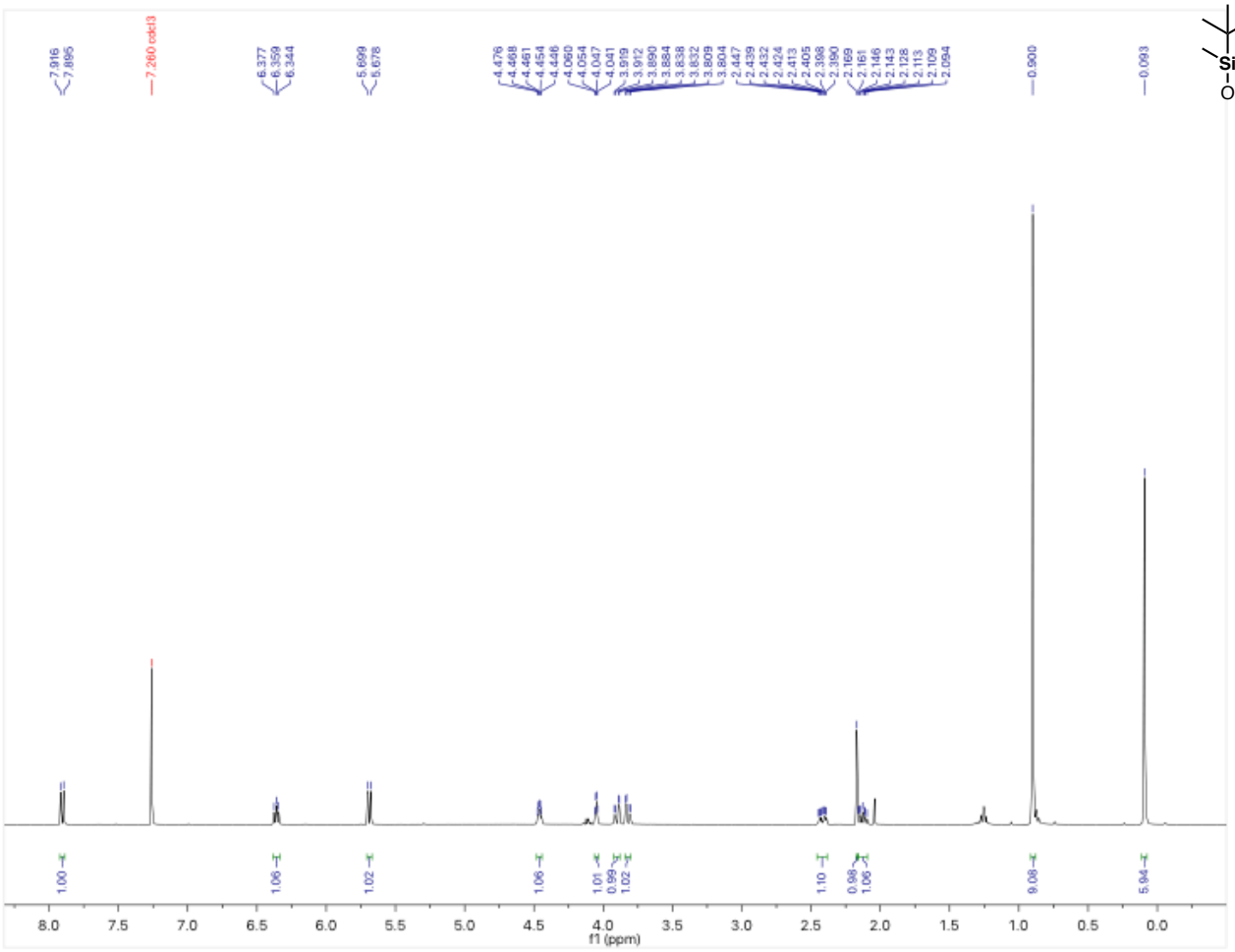

160.69
157.30
137.06
120.66
120.02
77.32 cdcl3
77.07 cdcl3
76.81 cdcl3
62.35
61.91
35.63
31.80
31.55
24.07
f1 (ppm)

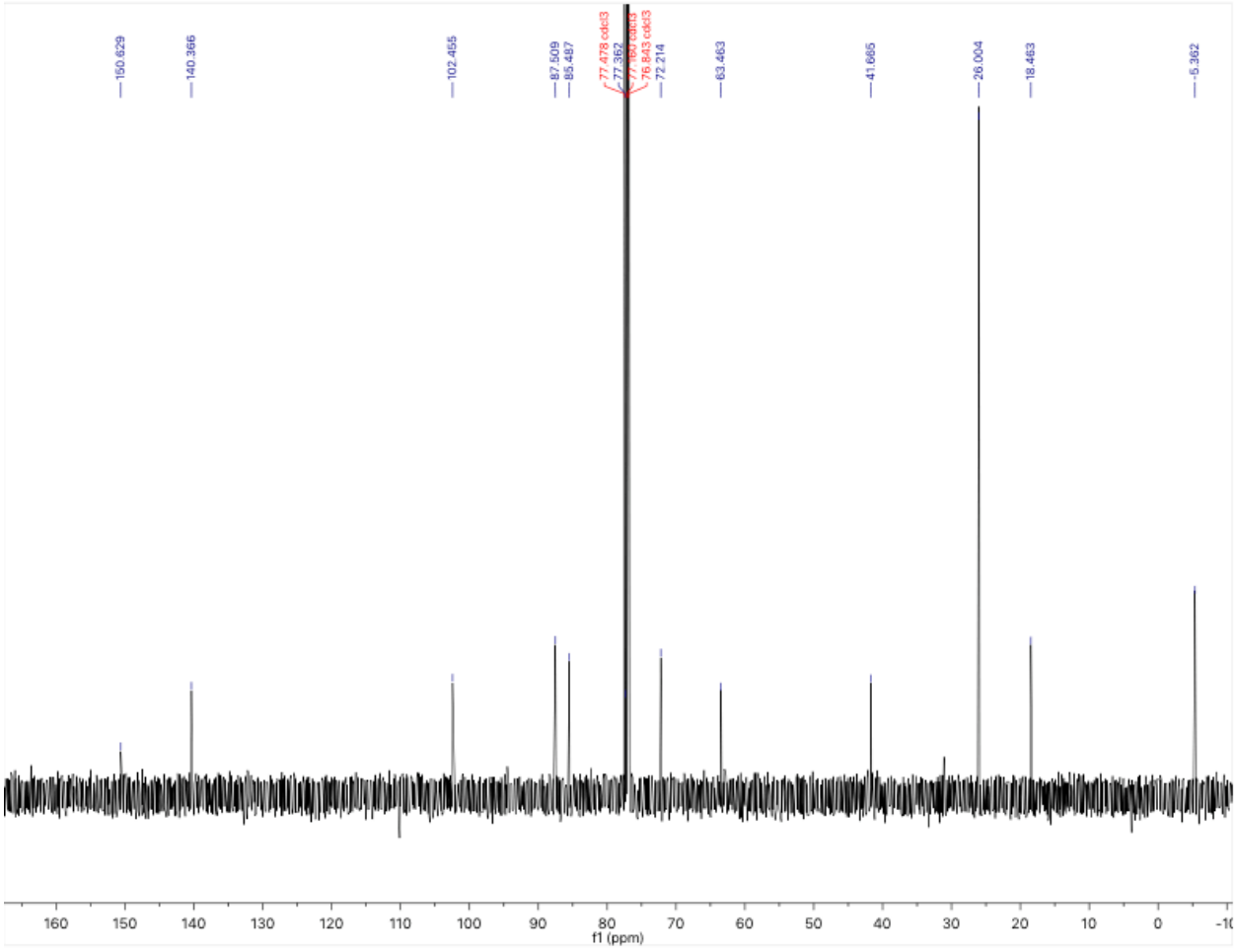

OH


7.240 cdcl3
4.955
4.953
4.950
4.939
4.936
4.933
4.866
4.863
4.860
4.857
4.854
4.849
4.847
4.844
4.841
3.779
3.766
3.756
3.743
3.617
3.601
3.567
3.550
3.544
3.527
1.710
1.707
1.683
1.681
1.674 HDO
1.656
1.653
1.646 HDO
1.639 HDO
1.365
1.348
1.183
1.167
1.137
1.134
1.127
1.112
1.106
1.104
1.096
1.085
1.075
1.060
1.042
1.040
1.033
1.027
1.025
1.018
1.010
0.842
0.831
0.828
0.825
0.818
0.814
0.812
0.802
0.50
0.82
0.94
2.00
8.70
0.51
3.00
1.79
2.88
2.03
0.87
f1 (ppm)


135.165
133.189
123.544
119.186
77.413 cdcl3
77.160 cdcl3
76.906 cdcl3
63.710
60.614
35.271
31.177
28.960
28.776
26.311
25.879
25.709
22.795
22.426
21.465
20.908
18.550
18.361
15.583
f1 (ppm)


O
NH
N
O
Si
O
O
OH


7.916
7.895
7.260 cdcl3
6.377
6.359
6.344
5.699
5.678
4.476
4.468
4.461
4.454
4.446
4.060
4.054
4.047
4.041
3.919
3.912
3.890
3.884
3.838
3.832
3.809
3.804
2.447
2.439
2.432
2.424
2.413
2.405
2.398
2.390
2.169
2.161
2.146
2.143
2.128
2.113
2.109
2.094
0.900
0.093
1.00
1.06
1.02
1.06
1.01
0.99
1.02
1.10
0.98
1.06
9.08
5.94
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


160.629
140.366
102.455
87.509
85.487
77.478 cdcl3
77.362
77.160 cdcl3
76.843 cdcl3
72.214
63.463
41.665
26.004
18.463
-5.362
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)